

Evaluation of Multiple and Single Emission Peak Light Emitting Diode Light Curing  
Units Effect on The Degree of Conversion and Microhardness of Resin-based Pit and  
Fissure Sealant.

By

Saleh Ali M Alqahtani

Submitted to the Graduate Faculty of the School of Dentistry in partial fulfillment of the  
requirements for the degree of Master of Science in Dentistry, Indiana University School  
of Dentistry, 2017

Thesis accepted by the faculty of the Department of Operative Dentistry, Indiana University School of Dentistry, in partial fulfillment of the requirements for the degree of Master of Science in Dentistry.

---

Armando E. Soto

Chair of the Research Committee

---

Jeffrey A. Platt

Research Committee Member

---

Norman Blaine Cook

Program Director

---

Date

## DEDICATION

All the praises to my God whose grace sustains.

This thesis is dedicated to all the people who support me in my life:

To the souls of my greatest father and mother.

To the most fabulous family my wife, sons, and daughter.

To my lovely brothers and sisters for their love and encouragement during my studies.

To my friends, who were like my second family during my study period especially Naif Nabil.

## ACKNOWLEDGMENTS

I would like to convey my deepest gratitude to King Khalid University for giving me the opportunity to continue my graduate studies.

I would like to express my respect and appreciation to my mentor, Dr. Armando E. Soto.

I thank him for his valuable guidance, knowledge, and efforts.

I also would like to send my thanks to my program director, Dr. Norman Blaine Cook, and the research committee member Dr. Jeffrey Platt for his helpful suggestions during the experimental phase of the project.

My sincerest gratitude goes to Dr. Afnan Al-Zain for her knowledge and support that guided me to finish my research.

Finally, I would like to thank also the statisticians, George Eckert and Tang Qing for their help.

## TABLE OF CONTENTS

Introduction.....	1
Review of Literature.....	8
Methods and Materials.....	19
Results.....	24
Tables and Figures.....	29
Discussion.....	45
Summary and Conclusions.....	51
References.....	53
Abstract.....	65
Curriculum Vitae	



## LIST OF TABLES AND ILLUSTRATIONS

TABLE-1	Details of the composition of resin-based sealant and light-curing units used in the study as described by the manufacturers.	30
TABLE -2	Tested groups identified by light, distance and time.	31
TABLE -3	The randomized sequence of group preparation and testing.	31
TABLE -4	Mean (standard deviation) irradiance values ( $\text{mW}/\text{cm}^2$ ) of the top surface for each light curing unit at the different light curing distances and curing times.	32
TABLE -5	Mean (standard deviation) irradiance values ( $\text{mW}/\text{cm}^2$ ) of the bottom surface for each light curing unit at the different light curing distances and curing times.	32
TABLE -6	Mean (standard deviation) for the degree of conversion values of the top surface of the resin-based sealants cured by each light-curing unit explored at the different curing distances and curing times.	33
TABLE -7	Mean (standard deviation) for the degree of conversion values on the bottom surface of the resin-based sealants cured by each light-curing unit explored at the different curing distances and curing times.	33
TABLE -8	Mean (standard deviation) microhardness values (Knoop Hardness Number) on the top surface of the resin-based sealants cured by each light-curing unit explored at the different curing distances and curing times.	34
TABLE -9	Mean (standard deviation) microhardness values (Knoop Hardness Number) on the bottom surface of the resin-based sealants cured by each light-curing unit explored at the different curing distances and curing times.	34
TABLE -10	Microhardness bottom/top ratio.	35
TABLE -11	Mean (standard deviation) Radiant exposure – Top ( $\text{J}/\text{cm}^2$ ).	35
TABLE-12	Mean (standard deviation) Radiant exposure – Bottom ( $\text{J}/\text{cm}^2$ ).	35

FIGURE-1	Managing Accurate Resin Curing System-Resin Calibrator (MARC-RC) system, top (on the right) and bottom (on the left) sensors.	36
FIGURE -2	Mechanical arm used to center The LCU light guide tip on the MARC-RC top sensor.	37
FIGURE -3	Reference points on the MARC-RC system, the rims of LCUs and transparent guide template were used to standardize the positions of LCUs throughout the measuring process.	38
FIGURE -4	Marks on the mold were placed to standardize the location of the sample when performing the DC and microhardness experiments.	39
FIGURE -5	Sample mold filed with resin-based pit and fissure sealant after that was placed between tow glass slabs to avoid air entrapment.	40
FIGURE -6	The LCU light guide tip on the MARC-RC bottom sensor.	41
FIGURE -7	Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) spectroscopy device.	42
FIGURE -8	The crystal plate and samples secured using a swivel pressure clamp on the FTIR-ATR to stabilize the sample on the crystal.	43
FIGURE-9	Five indentations were located in the upper, lower, left, right, and center of each test surface with the indentations 1mm from the periphery and 2mm between indentations.	44
CHART-I	Experiment flow chart	44

## INTRODUCTION

The National Health and Nutrition Examination Survey 2011-2012 data indicated that, in the United States, about one-fourth of children and more than one-half of adolescents experienced dental caries in their permanent teeth.<sup>1</sup> Occlusal surfaces, particularly those on permanent molars, have grooves called pits and fissures that can trap debris and microorganisms, thus increasing the risk of developing dental caries lesions.<sup>2</sup> A pit and fissure sealant is a relatively low viscosity resin material that is applied to the occlusal pits and fissures of caries susceptible teeth and then polymerized, either chemically (auto polymerizing) or by exposing it to visible light (light-cured).<sup>3</sup> This forms a micromechanically bonded protective layer that prevents the penetration of bacterial products and cuts off the access of surviving caries producing bacteria from their source of nutrients. Pit and fissure sealants were introduced in the 1960s. Buonocore's publication in 1955 was the commencement of the acid-etch technique in dentistry. Sealing pits and fissures with resin to prevent caries was the first clinical application of the acid-etch technique.<sup>4-6</sup>

Currently, there are two types of pit and fissure sealant materials available: resin-based and glass ionomer cement. The resin-based sealants are further divided into generations according to their mechanism of polymerization or their content. The development of sealants has progressed from the first generation sealants that had to be activated with ultraviolet light, through the second and third generations of auto polymerized and visible light activated sealants, to the fourth generation containing fluoride. The first generation sealants are no longer marketed.<sup>1,7</sup> Commercially available sealants are classified according to the filler loading (free of inert fillers or semi-filled) or opacity (clear, tinted or opaque). Light activated resin-based materials with

camphorquinone (CQ)–tertiary amine initiating systems are the most commonly used. There exist other photoinitiator.<sup>8</sup>

Different types of light emitting diode (LED) light-curing units (LCUs) are available. The narrow emission spectrum of single emission peak LED LCUs is limited to 420–490 nm to match the narrow absorption peak of CQ (465 to 470 nm) in the blue wavelength range. This may lead to an insufficient capacity of such LCUs, irrespective of their light intensity, to cure resin-based materials containing photoinitiators other than CQ.<sup>9</sup>

These other photoinitiators have an absorption spectrum within the near ultraviolet region that extends to the violet visible light spectrum (380-420 nm) with a narrow absorption peak (395-410 nm).<sup>10</sup> In an attempt to overcome the problem of emission absorption mismatch of alternative photoinitiator containing materials, multiple emission peak LED LCUs were introduced with two narrow peaks in the range between 395–510 nm to match the absorption spectrum of CQ and the alternative photoinitiators. The latest generations of LED curing lights have shown better performance when compared to other LCUs.<sup>11,12</sup> However, the light delivery from the curing tip still has a significant effect on the polymerization; there is a significant decrease in irradiance as the distance from the curing light tips increases.<sup>12</sup>

Radiant exposure ( $\text{J}/\text{cm}^2$ ), sometimes incorrectly termed "energy density," is the total amount of energy delivered to a resin-based materials surface during the entire irradiation procedure. It is the product of light irradiance ( $\text{mW}/\text{cm}^2$ ) and irradiation time (s).<sup>13</sup> Some studies support the fact that radiant exposure is the primary determining factor of the material properties. The researchers assumed that the degree of conversion (DC) of a composite is directly proportional to the length of light exposure. Therefore, it is rational

to investigate the shortest curing time that provides the highest DC without deleteriously affecting the physical properties of the resin-based material.<sup>14</sup>

The degree of conversion (DC) is the percentage of double carbon links (C=C) present in the monomers that are converted to single links (C-C) to form the polymeric chain during the polymerization process. Among several methods to determine DC of resin-based materials, the Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR) has been demonstrated to be a dependable method as it detects the C=C stretching vibrations directly before and after curing of resin-based materials. A high percentage of DC is required to achieve good mechanical properties such as hardness,<sup>15,16</sup> flexural strength and wear resistance.<sup>17</sup> Hardness testing is a reliable method to test how well a resin is cured by testing the mechanical properties of the material. The Knoop microhardness (KHN) test has been shown to be one of the best methods for testing the hardness of resin-based materials, and a good correlation between the degree of conversion and the Knoop microhardness has been reported.<sup>18</sup> Microhardness gives an indication of the DC of the material. Measuring the microhardness bottom/top ratio of samples may provide information about the polymerization effectiveness where the ratio should be no more than 20% difference between the hardness values of top and bottom surfaces. However, some researchers disagree with this relationship as a rule because factors other than DC, like the degree of crosslinking may affect the microhardness. The investigators in previous studies suggest that the microhardness values do not provide quantitative information on the actual change in reactive groups.<sup>18</sup>

Although placing a dental sealant is a routine procedure, it is very technique sensitive. Attention to placement details, tooth isolation and curing light position may

diminish the need to repair/replace the dental sealant. As an example, the SEAL Indiana program at IUSD has placed over 35,000 dental sealants (DS) since its inception in 2003. However, many of the sealants placed are replacements. For example, during the period from 2007-2009, 834 children were evaluated at least twice and 940 DS placed, and 518 (61%) of these DS were either repaired or replaced. On the other hand, a meta-analysis concluded that Light-polymerizing resin-based sealants retention percentage after a 2-year observation period, 77.8% of the sealants remained intact and dropped to 73.3% after 5 years.<sup>19</sup> Therefore, besides the requisite of appropriate wavelength to activate the photoinitiators in sealant material, previous studies showed that sufficient intensity is also required for successful polymerization.<sup>83</sup> The fillers, opacity, and thickness of the materials affect light penetration. Moreover, the distance of the light curing tip from the surface and exposure duration are critical for the degree of conversion and can be influenced or controlled by clinicians to some extent. Ensuring sufficient curing is an integral requirement for the success and longevity of a pit and fissure sealant.<sup>20,21</sup> Insufficient polymerization of the polymer matrix may make resin-based fissure sealant material more sensitive to the plasticizing effects of exogenous substances which contain a variety of chemicals (e.g. acids, bases, salts, alcohols, oxygen, etc.). These substances are entering the oral environment during eating and drinking and may have a degrading effect on the polymer network and compromise its clinical longevity.<sup>21</sup>

The higher surface hardness and DC of a resin-based material, the better its clinical performance. Laboratory studies analyze different properties of resin-based fissure sealants such as the DC and surface hardness to determine the effectiveness of multiple emission peak or single emission peak LED LCUs with various distances and different curing times.



However, these laboratory studies are not able to simulate the clinical situation completely. More scientific evidence on the monomer to polymer conversion in commercial resin-based fissure sealants cured with single and multiple emission peak LED LCUs may help clinicians decide whether a multiple or single emission peak LED LCUs should be used for an efficient and predictable clinical performance for this type of fissure sealant.<sup>22-25</sup> Therefore, the aim of the present study was to assess a multiple emission peak light-emitting-diode (LED) light-curing unit (LCU) by measuring the polymerization efficiency through the degree of conversion (DC) and Knoop microhardness (KHN) of a resin-based pit and fissure sealant at various light curing times and two different distances compared to a single emission peak LED LCU.

## HYPOTHESES

### Null Hypotheses

1. The radiant exposure and irradiance delivered to the top and bottom of a resin-based pit and fissure sealant sample using a multiple emission peak LED LCU will not demonstrate significant differences compared to a single emission peak LED LCU at various curing times and multiple distances.
2. The degree of conversion of a resin-based pit and fissure sealant using a multiple emission peak LED LCU will not demonstrate significant differences compared to a single emission peak LED LCU at various curing times and multiple distances.
3. The microhardness of a resin-based pit and fissure sealant using a multiple emission peak LED LCU will not demonstrate significant differences compared

to a single emission peak LED LCU at various curing times and multiple distances.

#### Alternative Hypotheses

1. The radiant exposure and irradiance delivered to the top and bottom surfaces of a resin-based pit and fissure sealant sample using a multiple emission peak LED LCU will significantly increase compared to a single emission peak LED LCU with increasing the curing time and decreasing the distance.
2. The DC of a resin-based pit and fissure sealant using a multiple emission peak LED LCU will significantly increase compared to a single emission peak LED LCU with increasing the curing time and decreasing the distance.
3. The microhardness of a resin-based pit and fissure sealant using a multiple emission peak LED LCU will significantly increase compared to a single emission peak LED LCU with increasing the curing time and decreasing the distance.

## REVIEW OF LITERATURE

From a primary prevention perspective, anatomic grooves or pits and fissures on occlusal surfaces of permanent molars trap food debris and promote the presence of bacterial biofilm, thereby increasing the risk of developing caries lesions. Efficiently sealing these surfaces with a dental material, for example, pit-and fissure sealants can prevent caries lesions. This is part of a comprehensive caries preventive management approach.<sup>25</sup> From a secondary prevention perspective, there is evidence that sealants also can inhibit the progression of noncavitated caries lesions. The use of sealants to prevent the progression of caries lesions is critical to the clinician when determining the appropriate intervention for noncavitated caries lesions.<sup>27</sup>

Pit and fissure sealants are classified into two types of sealant materials: resin-based sealants and glass ionomer (GI) cement. Currently, resin-based sealants are the most commonly used. Resin-based sealants contain urethane dimethacrylate (UDMA), or bisphenol A-glycidyl methacrylate (bis-GMA) monomers polymerized by either a chemical activator and initiator or by light of a particular wavelength and intensity. Resin-based sealants are supplied as unfilled, colorless or tinted transparent materials or as filled, opaque, tooth-colored, or white materials.<sup>2,26</sup> The resin-based sealants are further divided into generations according to their mechanism of polymerization or their content. The development of sealants has progressed from the first generation sealants that were activated with ultraviolet light, through the second and third generations of auto polymerized and visible light activated sealants, to the fourth generation containing fluoride. First generation sealants are no longer marketed.<sup>1,7,8</sup> Light activated resin-based materials with camphorquinone (CQ)-tertiary amine initiating systems are the most commonly used.<sup>28</sup>

The first dental curing light was developed in the 1970s. It was the Nuva Light (produced by Dentsply/Caulk) and used ultraviolet light (UV). The use of UV light was discontinued because of adverse biological effects and poor penetration through tooth structure, and was replaced by visible blue light activated systems.<sup>26</sup> During the early 1980s progress in the area of visible light occurred. The Quartz–tungsten–halogen (QTH) bulb was introduced, which replaced the UV curing light.<sup>29</sup> The plasma arc curing light was introduced in 1998.<sup>30</sup>

When comparing commercially available blue LEDs with conventional QTH lamps, the LEDs curing lights have a narrow spectral range, require less operating power, generate less heat, cause less gingival/pulpal irritation, have long lasting bulbs and are often cordless featuring rechargeable batteries.<sup>31</sup> One study showed that the emission peak of a blue LED chip is at 465 nm and that it coincided with the absorption peak of CQ at 467 nm, although, early generation LED lights sometimes did not perform well compared to the QTH when the material contained a photoinitiator that absorbs light at lower wavelengths than CQ.<sup>32-34</sup> Manufacturers are currently promoting and selling newer generation LED lights with high power/high-intensity modes and a wider range of wavelengths.<sup>35</sup> The narrow emission spectrum of commercially available single emission peak LED LCUs is limited to 420–490 nm to match the narrow absorption peak of CQ (465 to 470 nm) in the blue wavelength range. However, the alternative photoinitiators have an absorption spectrum within the near ultraviolet region that extends to the violet visible light spectrum (380-420 nm) with a narrow absorption peak (395-410 nm).<sup>10</sup> In attempt to overcome the problem of emission absorption mismatch of alternative photoinitiator containing materials, multiple emission peak LED LCUs have been introduced with two

narrow peaks in the range between 395–510 nm to match the absorption spectrum of the CQ and the alternative photoinitiators.<sup>11,12</sup>

The resin matrix of the resin-based pits and fissures sealant material is usually composed of aromatic or aliphatic dimethacrylates monomers. Adequate light activation transforms the monomers into a complex polymer structure. Monomer conversion into polymers does not attain 100% as some monomer remains unreacted.<sup>36</sup> The polymerization process starts by absorbing the light at a specific range of wavelength. Once the photoinitiator is activated it reacts with the reducer agent (aliphatic amine) to produce free radicals. During polymerization, double carbon links (C=C) present in the monomers are converted into single links (C-C) to form a polymeric chain. The extent to which monomers react to form the polymer during the polymerization reaction has a substantial effect on the physical and mechanical properties of composites resins.<sup>21,37</sup>

Conventionally, the extent of polymerization is quantified by comparing the amount of remaining double bonds in the polymer structure to the initial amount. This ratio is expressed in percentage (%) and termed the DC.<sup>38</sup> The DC values vary for a broad range of resin-based material types 35–77%.<sup>39</sup> Following the initiation of photopolymerization, the degree of conversion and cross-linking density increase rapidly resulting in a rapid growth of the system viscosity and the first change of state, from a viscous liquid to an elastic gel, called gelation.<sup>40</sup> At this point, the mobility restriction mostly affects radicals, located on large molecules (growing polymer chains), whereas small monomer molecules can still diffuse readily. Consequently, bimolecular termination decreases dramatically while new growth centers are still created by initiation.<sup>38</sup> When the free radical concentration increases, this leads to a rapid increase in the rate of polymerization ( $R_p$ , a

fraction of double bond converted per second, representing the speed of the reaction) called auto-acceleration.<sup>41</sup> As the reaction proceeds, the viscosity increases limiting diffusion even for monomer molecules, resulting in significant decrease of  $R_p$ . This effect corresponds to the second change of state, from rubbery to glass, or vitrification.<sup>42</sup> Vitrification prevents any further extensive reaction and explains why DC cannot reach 100%, even with optimum irradiation conditions.<sup>43</sup>

Several methodologies can be used to evaluate the photopolymerization efficiency. The most common technique used is DC which is significantly correlated to the mechanical properties,<sup>44,45</sup> volumetric shrinkage,<sup>46</sup> wear resistance<sup>47</sup> and monomer elution.<sup>48</sup> It is measured most commonly by spectroscopic techniques that infer the quantity of remaining double bonds, using either an infrared Fourier transform spectroscopy (FTIR) or a Raman spectroscopy.<sup>49,50</sup> A higher DC in a resin system provides increased mechanical properties that, in turn, should improve the restoration's longevity.<sup>50</sup>

The microhardness of a resin-based material has shown to correlate with the degree of monomer conversion. The Knoop hardness number (KHN) predicts the relative DC for a particular resin under variable conditions. Since the impact of the light source is not well known, investigations are needed to examine the relationship using microhardness bottom/top ratios (KHN B/T ratio).<sup>51</sup> The DC has been indirectly evaluated by microhardness measurements (ether Vickers or Knoop microhardness) as a good linear correlation was observed between the DC and the microhardness values.<sup>52,53</sup> However, some researchers disagree with this relationship as a general rule because factors other than the DC, such as the degree of crosslinking, may affect microhardness.<sup>54</sup> In any case, microhardness measurement does not provide quantitative information on the actual

change in reactive groups.<sup>38</sup> Moreover, other properties like the degree of crosslinking,<sup>55</sup> mechanical properties,<sup>56,57</sup> shrinkage and shrinkage stress,<sup>44</sup> depth of cure,<sup>58</sup> trapped free radicals<sup>59</sup> and biocompatibility<sup>16</sup> can be used as indirect evaluation methods.

Photopolymerization efficiency may be affected by intrinsic or extrinsic factors. The type and concentration of the photoinitiator systems have a significant impact on the polymerization process and are considered intrinsic factors.<sup>60,61</sup> There exist a high correlation between the increase in the DC and hardness with increased photoinitiator concentration.<sup>62,63</sup> When CQ/amine levels are increased beyond optimum value, a reduction of DC and hardness was observed. This may be due to more absorption of light in the top regions, resulting in less light transmission to the bottom layers.<sup>62</sup> Furthermore, using the ternary photoinitiator systems, such as combining iodonium salts with CQ/amine, results in an increase in DC, degree of cross-linking, mechanical properties and color stability.<sup>64-66</sup>

Viscosity, monomer and filler type are intrinsic factors affecting the photopolymerization efficiency. There is evidence that initial resin viscosity is a significant element in the reaction kinetics and final DC of dimethacrylate polymers as it affects the mobility of each monomer and its reactivity.<sup>42</sup> Variations of monomer molecular structure (di- or polymethacrylates, molecular weight, molecule stiffness, etc.) and proportions can significantly affect the polymerization efficiency.<sup>38</sup> For pure bis-GMA, the maximum polymerization is less than 5% of conversion due to the very high viscosity, and the final DC is limited to about 30%. In contrast, for pure triethylenglycol dimethacrylate (TEGDMA), which is far less viscous, the maximum rate is observed around 22% of conversion, with a final DC of over 60%, while the different co-monomer mixtures in the



system show intermediate values between these two extremes.<sup>67</sup> Moreover, the filler content can affect the polymerization process of resin-based material as well.<sup>38</sup> One study found that the filler volume in the resin formulation in addition to the differences in fillers size and geometry resulted in significant differences in DC, from 48 to 61%.<sup>68</sup>

Lastly, the optical properties of a resin composite and their photopolymerization reaction are interdependent.<sup>38</sup> Several factors can limit the light transmission through the resin-based material. First, light reflection occurs at the surface.<sup>69,70</sup> Second, light is absorbed, either by pigments<sup>71</sup>, (which explains the lower depth of cure observed for darker and more opaque shades) or by photo-initiators.<sup>89</sup> Moreover, the filler particle dimensions can affect the light transmission by scattering, which depends on the particle size and the incident wavelength of the curing light.<sup>68,72</sup>

Extrinsic factors also effect photopolymerization efficiency. Light curing units and their emission spectrum are some extrinsic factors affecting efficiency of photopolymerization of the resin. Different types of light curing units have been used in the photopolymerization process with different effectiveness as that discussed previously.<sup>73-79</sup> The first generation LED was commercially available by the end of the year 2000.<sup>79</sup> This technology emitted only blue light with wavelengths between 440-480 nm without filtering.<sup>26</sup> Many advantages of LED over other light curing units include: low wattage, battery powered, no heat generation and no fan noise. The most recent LED generation combines two or more LED chip types to increase intensity and to extend the range of the wavelengths.<sup>26</sup> These LED curing lights have shown outstanding performance when compared to other types of curing light units.<sup>80</sup> However, light delivery from the curing tip still has a significant effect on the polymerization.<sup>40</sup> Well-collimated straight light guides

have helped in reducing the amount of light wasted by focusing the rays, thus increasing the light delivery.<sup>81</sup> In addition, there is concern regarding the heterogeneity of the cure over the surface which can be produced from the use of an array of several diodes or from the way the light is transmitted through the tip.<sup>81, 82</sup> The uniformity of the light intensity can be improved, for example by using additional optical elements (mixing tube and diffusing screen).<sup>83</sup> LED lights are more efficient than broad spectrum halogen lights to polymerize CQ-based materials because the spectrum of blue LED lights is centered on the CQ maximum absorption peak, thus reducing the cure time. However, other photoinitiator systems may require broad-spectrum lights.<sup>17,84</sup>

Other extrinsic factors such as radiant exposure, irradiance and irradiation time also impact photo-polymerization efficiency. Radiant exposure ( $\text{J}/\text{cm}^2$ ) is the total amount of energy delivered to a resin-based materials surface. It is the product of time and light irradiance ( $\text{mW}/\text{cm}^2$ ) which is defined as the power of electromagnetic radiation per unit area. The radiant exposure is considered the main determining factor of the material properties.<sup>85,86</sup> Localized differences in irradiance and wavelength distribution can have a significant impact on the relevance of measurements made to describe the properties of light-cured resin-based materials.<sup>82,83,87-91</sup> Some regions across the light tip may deliver high irradiance, and others may provide low irradiance with an entirely different spectral emission. Thus, if any inhomogeneity is present in the light beam, the resin-based material will not receive the average irradiance or spectral emission from the LCU and may produce misleading results. Although it is recognized that there would be some dispersion of the light by the resin-based material, somewhat mitigating the effect of beam inhomogeneity,

the beam profile has been reported to be mirrored in the microhardness distribution across the resin-based light cured material.<sup>83, 88, 89</sup>

Irradiation modes are extrinsic factors that may affect photo-polymerization efficiency. Different “soft-start” curing protocols (ramp, step or pulse-delay modes) were proposed. Soft start curing modes were introduced to provide a low initial rate of polymerization to delay the onset of polymer gelation and thus reducing the polymerization shrinkage stresses. However, others observed that a soft-start regimen had the potential to reduce shrinkage stress while keeping the DC and the mechanical properties constant.<sup>92-95</sup> Despite a substantial number of publications on this subject, there is still no definitive answer as to whether or not soft-start modes are beneficial. Again, this is probably due to the differences in the composition of the various resin-composites used in the different studies, which probably affect the efficiency of the soft-start curing modes and the resulting properties.

Moreover, temperature plays a significant role in the polymerization reaction. The change in temperature from room temperature (22 °C) to the mouth temperature (35 °C) have shown to increase hardness, polymerization rate, and DC (6–10%).<sup>96</sup> The increase in temperature allows more monomer mobility hence, more of the reaction occurs before vitrification.<sup>97</sup> To avoid any error in studies involving resin-based materials, temperature stability should be considered.

Clinically, the orientation and positioning of the LCU may have a dramatic effect on both the irradiance and wavelength received by different locations in the restoration.<sup>98</sup> This can be considered as an effective extrinsic factor on photo-polymerization efficiency. The position of the guide tip of the LED LCU affects the amount of energy delivered to

the resin-based material and significantly influences polymerization efficiency.<sup>38</sup> There is a decrease in irradiance when the distance increases between the restoration surface and the guide tip of the LCU.<sup>99,100</sup> Therefore, manufactures recommend placing the tip as close as possible to the resin surface being cured. Also, keeping the light guide stable and in a perpendicular position to the restoration helps to ensure polymerization depth.<sup>101</sup>

In conclusion, factors such as exposure time, monomer composition, and opacity of the material have significant effects on the depth of curing of the resin-based material. The materials' DC is proportional to the amount of light to which they are exposed. Fissure sealants are commercially available in different types regarding the opacity for instance; translucent, yellowed, or opaque. Opaque resin-based sealant materials have shown less light absorption and transmittance to the bottom surface of the material; therefore, short curing times may provide insufficient depth of cure. To ensure optimal polymerization, dental professionals have the responsibility to select the type of materials, the LCU that are optimally matched and an effective curing time. There are a wide variety of light cured resin-based materials and LCUs available in the market. The selection of the LCU is intimately connected with the material's characteristics which are often unknown or unclearly identified by clinicians or manufacturers. Even if the exact product composition was known, many other factors could affect a given material property, making it impossible to predict the resulting polymerization quality. Therefore, there is a critical need for better information from manufacturers on their products to be able to adapt and optimize the use of resin-based materials in the daily practice. For each new material appearing on the market, it would be beneficial if essential information, such as the absorption spectrum,

and the impact of various irradiance/time combinations on the principal material properties such as, the DC, hardness and material properties, were provided.

## MATERIALS AND METHODS

A laboratory study was conducted using an opaque resin-based pit and fissure sealant (Delton, DENTSPLY, York, PA). (Table-1) Two light curing units were evaluated, a multiple emission peak LED LCU (VALO, Ultradent, South, Utah) and a single emission peak LED LCU (FLASH LITE 1401, Discus Dental, Culver, CA). (Table-1)

## SAMPLE PREPARATION

The light irradiance of the LED LCUs was measured using the 4mm diameter top sensor of a Managing Accurate Resin Curing System-Resin Calibrator (MARC-RC) system (Blue light Analytics Inc., Halifax, Canada). (Figure-1) Irradiance of the LCUs was measured each time before testing, to ensure the irradiance delivered from the LCUs remained consistent throughout the study and to simulate the amount of irradiance and radiant exposure received on the top surface of the samples. A mechanical arm was used to mount both LCUs in the same position and distance during specimen light curing. (Figure-2) Reference points on the MARC-RC system, the rims of LCUs and transparent guide template were used to standardize the positions of LCUs throughout the measuring process. The LCU light guide tip was placed in perpendicular position and centered on the MARC-RC top, bottom sensors and the top surface of the specimens. (Figure-3) Due to the shape of the light guide of the multiple emission peak LCU, a custom metal ring was used to ensure the guide tip was perpendicular on the sensors. The single emission peak LCU guide tip was flat so it was adjusted on the sensors directly. A total of 60-disc samples were fabricated using a Delrin mold (Figure-4) (6mm x 1mm) and divided into twelve groups identified by light, distance and time (n=5/group). (Table-2) The sealant material was injected into the mold sandwiched between Mylar strips and microscope slides to create a

smooth surface and avoid air entrapment. (Figure-5) Each sample was placed over the 4mm diameter MARC-RC bottom sensor to establish the amount of light irradiance delivered to the bottom surface of the specimen. (Figure-6) Equally positioned markings on four corners of the mold were created to standardize the location of the sample in the MARC-RC. Samples were cured for 10, 20, or 40 seconds at a 2 or 4 mm distance from the tip of the light guide to the top of the sample. The samples were not removed from the mold so that markings on the mold could be utilized to standardize sample position during DC and microhardness measurements. For each group, the samples were fabricated and stored in a transparent plastic container with multiple compartments. Each sample was placed in a separate compartment with a specific number from one to five. Next, the samples were covered with a moist paper towel and the lid of the container closed to maintain 100% relative humidity. The container was wrapped in aluminum foil to keep specimens away from the light and stored at 37°C for one hour in the incubator. The DC test was then performed followed by the KHN microhardness test. (Chart-I) The test groups were randomized for specimen fabrication and testing. Only specimens from one group were fabricated and tested each day. (Table 3)

#### DEGREE OF CONVERSION (DC)

The DC of a resin-based fissure sealant was measured using an Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) Spectroscopy (JASCO 4100 International Co., Tokyo, Japan) using a 1.8 mm diameter Diamond crystal plate (ATR-FTIR MIRacle™, Pike technologies, Madison, WI, USA). (Figure-7) The absorbance was measured using 64 scans and 4 cm<sup>-1</sup> resolutions. Three uncured resin-based fissure sealant samples were



measured. For the cured samples (n=5/group), three non-overlapped standardized measurements on the top or bottom surfaces were collected on each sample; one on the upper half, lower right and lower left side of each sample. Every cured specimen was placed on the diamond crystal plate and secured with a swivel pressure clamp to insure its adaptation. (Figure-8) The mid spectral region of infrared was used (MIR – from 400 to 4,000 cm<sup>-1</sup>). The DC was determined by measuring the intensity (or area) decrease of the methacrylate aliphatic (C=C) stretch absorption band at 1,637 cm<sup>-1</sup> as the methacrylate monomer was converted to polymer. The present aromatic bands at 1,607 which were used as internal standards. The areas under the curves (1607 and 1637 cm<sup>-1</sup>) of uncured and cured resin-based pit and fissure sealant was used to calculate the DC percent according to the following equation:

$$\text{Degree of conversion} = \left( 1 - \frac{\text{cured (area under 1637/area under 1607)}}{\text{uncured (area under 1637/area under 1607)}} \right) \times 100$$

The average DC values were calculated for each surface.

#### KNOOP MICROHARDNESS (KHN)

On the same specimens prepared for the DC, the KHN test was performed. Five indentations were made on both the top and bottom surfaces of each specimen. Indentations were located in the upper, lower, left, right, and center of each test surface with the indentations 1mm from the periphery and 2mm between indentations. (Figure- 9) The indentation location was standardized according to the markings placed on the mold. The hardness testing was performed using a hardness tester (Leco LM247AT, MI, USA, software; Confident V 2.5.2), with a diamond indenter utilizing 25-gram load and 10 second dwell time. The average KHN values were calculated for each surface.

## STATISTICAL ANALYSIS

The effects of LED LCU type (multiple or single emission peak), curing time (10, 20 and 40 seconds), distance (2, 4 and 6 mm), and surface (top and bottom) on radiant exposure, irradiance, degree of conversion, and microhardness was examined using ANOVA. The ANOVA included fixed effects for the 4 factors and their interactions and a random effect to correlate the measurements from the top and bottom surfaces of the same sample. A standard 3-way ANOVA was performed for the bottom/top ratios. Pair-wise comparisons were made using Fisher's Protected Least Significant Differences to control the overall significance level at 5%. The distributions of the measurements were examined and a transformation of the data (e.g. logarithm, square root, etc.) may be used to satisfy the ANOVA assumptions.

## SAMPLE SIZE JUSTIFICATION

Standard deviations are estimated to be 3.0 for KHN and DC was based on prior studies Lucey 2014 and Borges 2011. Calculations assume two-sided tests conducted at a 5% significance level, and assume no interactions among the factors. With a sample size of 5 for each group, for 80% power to detect a KHN or DC difference of 1.8 between LED LCU types.

## RESULTS

## IRRADIANCE MEASUREMENTS FROM THE LCUs

On the top MARC-RC sensor:

ANOVA results in Table-4 reveals that there were statistically significant differences detected by the top MARC-RC sensor between both LCUs, among curing times and between distances. The irradiance at 2mm curing distance was significantly higher than 4mm curing distance for both LCUs and curing time combinations. Moreover, the irradiance at 10 and 20 seconds curing time was significantly higher than 40 seconds at 2mm curing distances for the multiple emission peak LED LCU. Furthermore, the irradiance of the single emission peak LED LCU was significantly lower than multiple emission peak LED LCU for all the curing time and distance combinations.

On the bottom MARC-RC sensor:

ANOVA results in Table-5 showed that there were statistically significant differences in irradiance measurements detected by the bottom MARC-RC sensors cured by both LCUs, among curing times and between the curing distances, reflecting the irradiance received at the bottom surfaces of the samples. The irradiance at 2mm curing distance was significantly higher than 4mm distance for both LCUs and all curing time combinations. Moreover, the irradiance at 10 and 20 second curing times were significantly lower than 40 second for both LCUs at 2mm distance. The irradiance of single emission peak LED LCU was significantly lower than the multiple emission peak LED LCU at all curing times and curing distances.

## DEGREE OF CONVERSION OF THE SEALANT MATERIAL

On the top surfaces of the samples:

ANOVA results in Table-6 showed that there were significant differences in degree of conversion at the top surfaces of the samples among the curing times and between the distances. The degree of conversion at 2mm curing distance was significantly higher than 4mm curing distance at 10 second curing time with the multiple emission peak LED LCU and the single emission peak LED LCU. Moreover, the degree of conversion at 10 second curing time was significantly lower than 20 and 40 second using both curing units at 4mm curing distance.

On the bottom surfaces of the samples:

ANOVA results in Table-7 displayed that there were significant differences in degree of conversion at the bottom surfaces of the samples between both LCUs, among the curing times and between the distances. The degree of conversion at 2mm curing distance was significantly higher than 4mm curing distance with both LCUs at 10 second and the single emission peak LED LCU at 20 second. In addition, the degree of conversion at time 10 second curing time was significantly lower than 20 seconds and 40 seconds curing time using both light curing units and both curing distances. The degree of conversion at time 20 second curing time was significantly lower than 40 second at 4mm curing distance for both light curing unit. Furthermore, the degree of conversion with the single emission peak LED

LCU was significantly lower than multiple emission peak LED LCU all curing times at 4mm curing distances and 2mm curing distance at 20 second curing time.

#### KNOOP MICROHARDNESS OF SEALANT MATERIAL

On the top surfaces of the samples:

ANOVA results in Table-8 revealed that there were significant differences in the knoop hardness number at the top surface of the samples between the curing units, curing times, and curing distances. The knoop hardness number at 2mm distance was significantly higher than 4mm curing distance for 10 second curing time for both light curing units. Moreover, the knoop hardness number at 10 second curing time was significantly lower than 20 seconds at 4mm curing distance and both curing units. The knoop hardness number values at 10 seconds curing time were significantly lower than 40 seconds for both distances and both light curing units. In addition, the knoop hardness number at 20 second curing time was significantly lower than 40 second for 2mm distances with the multiple emission peak LED LCU. The single emission peak LED LCU was significantly lower than multiple emission peak LED LCU for all curing times and both distances.

On the bottom surfaces of the samples:

ANOVA results in Table-9 showed that there were significant differences in the knoop hardness number at the top surface of the samples among the

curing units, curing times, and curing distances. The knoop hardness number at 2mm distance was significantly higher than 4mm distance for all curing times with single emission peak LED LCU and with 10 second curing time for multiple emission peak LED LCU. Moreover, the knoop hardness number values at 10 second curing time were significantly lower than 20 second and 40 seconds curing time. Similarly, at 20 second curing time was significantly lower than 40 seconds for both distances and both light curing units. Furthermore, the single emission peak LED LCU was significantly lower than the multiple emission peak LED LCU for 40 second curing time at both distances and for 20 second at 4mm curing distance.

## TABLES AND FIGURES



TABLE-1

Details of the composition of resin-based sealant and light-curing units used in the study as described by the manufacturers.

Material/unit	Product name (manufacturer)	Composition
Pits and fissures sealant	Delton, DENTSPLY, York, PA	Aromatic an aliphatic dimethacrylate monomers  Titanium Dioxide (opaque)  Silicon Dioxide (Opaque)  Initiators  Stabilizers
Light Curing Unit	single emission peak  LED (FLASH LITE 1401, Discus Dental, Culver, CA)	Wavelength Range: 460-480 nm  Light Intensity: $\geq 1100 \text{ mW/cm}^2$
	multiple emission peak  LED LCU (VALO, Ultradent, South, Utah)	Wavelength Range: 395–480nm  Light Intensity: Irradiance ( $\text{mW/cm}^2$ )  Standard Power: $1000 \text{ mW/cm}^2$  High Power: $1400 \text{ mW/cm}^2$  Xtra Power: $3200 \text{ mW/cm}^2$

TABLE-2  
Tested groups identified by light, distance and time.

LED LCU	Multiple emission peak LED LCU		Single emission peak LED LCU	
Distance (mm)	2	4	2	4
Curing time (sec.)				
10	V2-10	V4-10	F2-10	F4-10
20	* V2-20	V4-20	* F2-20	F4-20
40	V2-40	V4-40	F2-40	F4-40

n=5/group

\* Control groups

TABLE-3  
The randomized sequence of group preparation and testing.

1 <sup>st</sup> day	2 <sup>nd</sup> day	3 <sup>rd</sup> day	4 <sup>th</sup> day	5 <sup>th</sup> day	6 <sup>th</sup> day
2mm-V20	2mm-F10	2mm V10	V20-4mm	4mm-F20	4mm-F40
7 <sup>th</sup> day	8 <sup>th</sup> day	9 <sup>th</sup> day	10 <sup>th</sup> day	11 <sup>th</sup> day	12 <sup>th</sup> day
2mm-F20	4mm-V40	2mm-F40	4mm-V10	2mm-V40	4mm-F10

TABLE – 4  
Mean (standard deviation) irradiance values (mW/cm<sup>2</sup>) of the top surface for each light curing unit at the different light curing distances and curing times.

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2mm	4mm	2mm	4mm
10 sec	1300.8 (7.6) <sup>Aa*</sup>	904.9 (2.0) <sup>Ab*</sup>	768.3 (6.9) <sup>Aa</sup>	365.2 (1.0) <sup>Ab</sup>
20 sec	1312.6 (6.2) <sup>Aa*</sup>	911.1(7.7) <sup>Ab*</sup>	763.0 (3.3) <sup>Aa</sup>	361.3 (1.5) <sup>Ab</sup>
40 sec	1225.3 (14.3) <sup>Ba*</sup>	898.6 (6.8) <sup>Ab*</sup>	766.0 (5.2) <sup>Aa</sup>	360.7 (2.0) <sup>Ab</sup>

Different lowercase letters in each row and uppercase letters in each column indicate statistically significant differences in each LCU. \*represents significantly different values between LCUs at the specific time and distance.

TABLE – 5  
Mean (standard deviation) irradiance values (mW/cm<sup>2</sup>) of the bottom surface for each light curing unit at the different light curing distances and curing times.

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2mm	4mm	2mm	4mm
10 sec	171.0 (1.2) <sup>Ba*</sup>	119.1 (1.4) <sup>Ab*</sup>	93.4 (0.5) <sup>Ba</sup>	46.3 (0.5) <sup>Ab</sup>
20 sec	172.3 (3.2) <sup>Ba*</sup>	121.9 (1.5) <sup>Ab*</sup>	93.2 (1.5) <sup>Ba</sup>	47.3 (1.3) <sup>Ab</sup>
40 sec	200.2 (7.7) <sup>Aa*</sup>	121.4 (2.1) <sup>Ab*</sup>	97.8 (3.1) <sup>Aa</sup>	47.2 (1.1) <sup>Ab</sup>

Different lowercase letters in each row and uppercase letters in each column indicate statistically significant differences in each LCU. \*represents significantly different values between LCUs at the specific time and distance.

TABLE – 6

Mean (standard deviation) for the degree of conversion values of the top surface of the resin-based sealants cured by each light-curing unit explored at the different curing distances and curing times.

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2mm	4mm	2mm	4mm
10 sec	78.6 (7.2) <sup>Aa</sup>	64.0 (11.3) <sup>Bb</sup>	77.6 (3.7) <sup>Aa</sup>	64.6 (11.0) <sup>Bb</sup>
20 sec	79.0 (7.9) <sup>Aa</sup>	83.8 (2.4) <sup>Aa</sup>	82.3 (2.6) <sup>Aa</sup>	79.6 (4.6) <sup>Aa</sup>
40 sec	84.0 (2.2) <sup>Aa</sup>	81.8 (5.3) <sup>Aa</sup>	85.2 (2.3) <sup>Aa</sup>	76.6 (7.8) <sup>Ab</sup>

Different lowercase letters in each row and uppercase letters in the column indicates statistically significant differences in each LCU.

TABLE – 7

Mean (standard deviation) for the degree of conversion values on the bottom surface of the resin-based sealants cured by each light-curing unit explored at the different curing distances and curing times.

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2mm	4mm	2mm	4mm
10 sec	45.4 (2.8) <sup>Ba</sup>	27.7 (4.9) <sup>Cb*</sup>	45.7 (3.5) <sup>Ba</sup>	20.4 (2.9) <sup>Cb</sup>
20 sec	77.9 (2.6) <sup>Aa*</sup>	76.4 (3.2) <sup>Ba*</sup>	71.5 (4.0) <sup>Aa</sup>	56.5 (6.4) <sup>Bb</sup>
40 sec	79.2 (4.2) <sup>Aa</sup>	81.4 (1.5) <sup>Aa*</sup>	74.8 (3.7) <sup>Aa</sup>	73.6 (1.9) <sup>Aa</sup>

Different lowercase letters in each row and uppercase letters in each column indicate statistically significant differences in each LCU. \*represents significantly different values between LCUs at the specific time and distance.

TABLE – 8

Mean (standard deviation) microhardness values (Knoop Hardness Number) on the top surface of the resin-based sealants cured by each light-curing unit explored at the different curing distances and curing times.

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2mm	4mm	2mm	4mm
10 sec	20.0 (2.1) <sup>Ba*</sup>	14.9 (3.1) <sup>Bb*</sup>	17.6 (0.7) <sup>Ba</sup>	5.9 (1.9) <sup>Bb</sup>
20 sec	21.2 (1.0) <sup>Ba*</sup>	18.4 (2.1) <sup>Aa*</sup>	19.2 (3.2) <sup>Aa</sup>	16.6 (2.0) <sup>Aa</sup>
40 sec	23.0 (3.9) <sup>Aa*</sup>	21.5 (1.1) <sup>Aa*</sup>	19.6 (0.6) <sup>Aa</sup>	17.6 (2.1) <sup>Aa</sup>

Different lowercase letters in each row and uppercase letters in each column indicate statistically significant differences in each LCU. \*represents significantly different values between LCUs at the specific time and distance.

TABLE – 9

Mean (standard deviation) microhardness values (Knoop Hardness Number) on the bottom surface of the resin-based sealants cured by each light-curing unit explored at the different curing distances and curing times.

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2mm	4mm	2mm	4mm
10 sec	4.9 (0.6) <sup>Ca</sup>	0.0 (0.0) <sup>Cb</sup>	5.2 (2.4) <sup>Ca</sup>	0.0 (0.0) <sup>Cb</sup>
20 sec	11.8 (1.7) <sup>Ba</sup>	10.0 (2.1) <sup>Ba*</sup>	10.0 (1.7) <sup>Ba</sup>	4.8 (3.0) <sup>Bb</sup>
40 sec	19.9 (1.4) <sup>Aa*</sup>	16.5 (2.2) <sup>Ab*</sup>	16.9 (1.4) <sup>Aa</sup>	10.3 (1.1) <sup>Ab</sup>

Different lowercase letters in each row and uppercase letters in each column indicate statistically significant differences in each LCU. \*represent significantly different values between LCUs at the specific distance.

TABLE – 10  
Microhardness bottom/top ratio.

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2mm	4mm	2mm	4mm
10 sec	0.24	0.0	0.29	0.0
20 sec	0.64	0.47	0.52	0.28
40 sec	0.87	0.76	0.86	0.58

TABLE-11  
Mean (standard deviation) Radiant exposure – Top ( $\text{J}/\text{cm}^2$ ).

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2mm	4mm	2mm	4mm
10 sec	13.0 (0.3)	9.1 (0.1)	7.7 (0.5)	2.4 (0.0)
20 sec	26.3 (0.1)	18.2 (0.1)	15.3 (0.1)	7.2 (0.2)
40 sec	49.0 (1.0)	36.0 (0.1)	30.6 (0.2)	14.4 (0.1)

TABLE-12  
Mean (standard deviation) Radiant exposure – bottom ( $\text{J}/\text{cm}^2$ ).

	Multiple emission peak LED LCU		Single emission peak LED LCU	
	2mm	4mm	2mm	4mm
10 sec	1.7 (0.0)	1.2 (0.1)	1.0 (0.0)	0.5 (0.0)
20 sec	3.5 (0.0)	2.4 (0.2)	1.9 (0.1)	1.0 (0.0)
40 sec	8.0 (0.1)	4.9 (0.9)	4.0 (0.1)	1.9 (0.1)

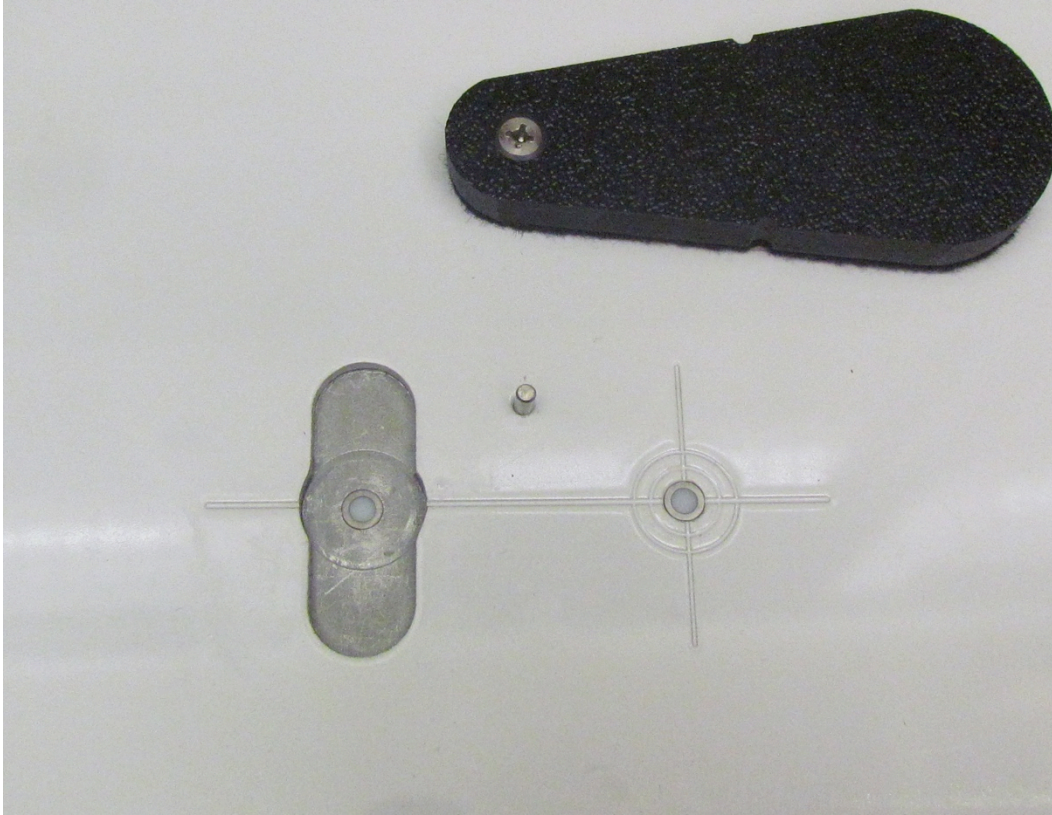


FIGURE-1. Managing Accurate Resin Curing System-Resin Calibrator (MARC-RC) system, top (on the right) and bottom (on the left) sensors.

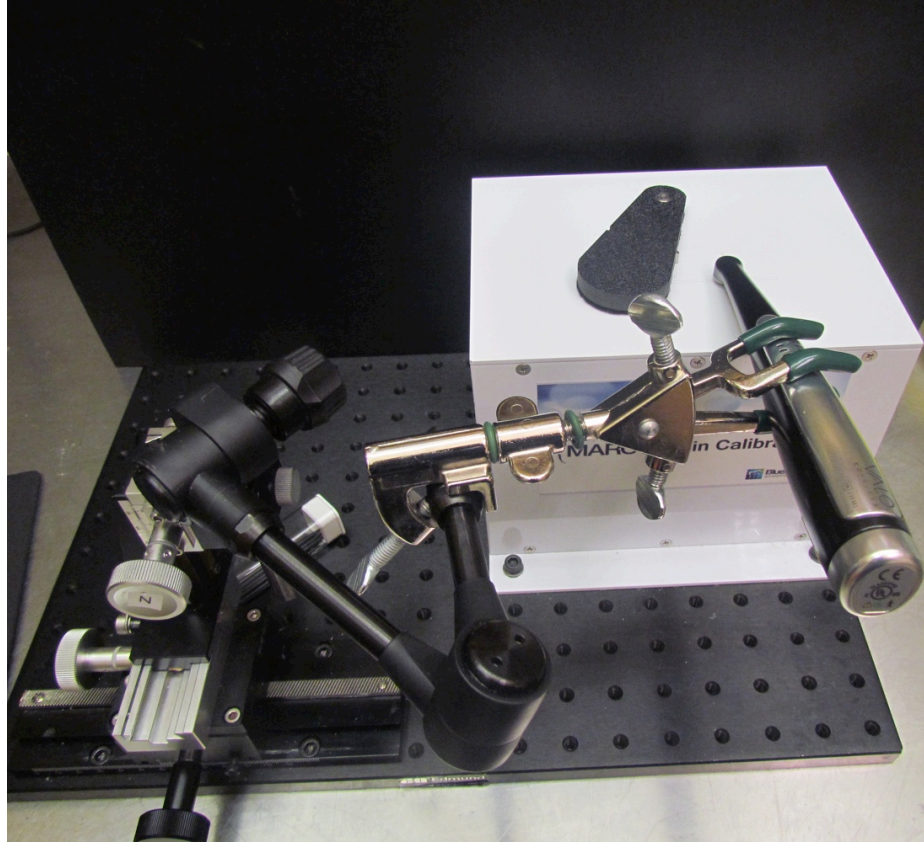


FIGURE-2. A mechanical arm used to center The LCU light guide tip on the MARC-RC top sensor.



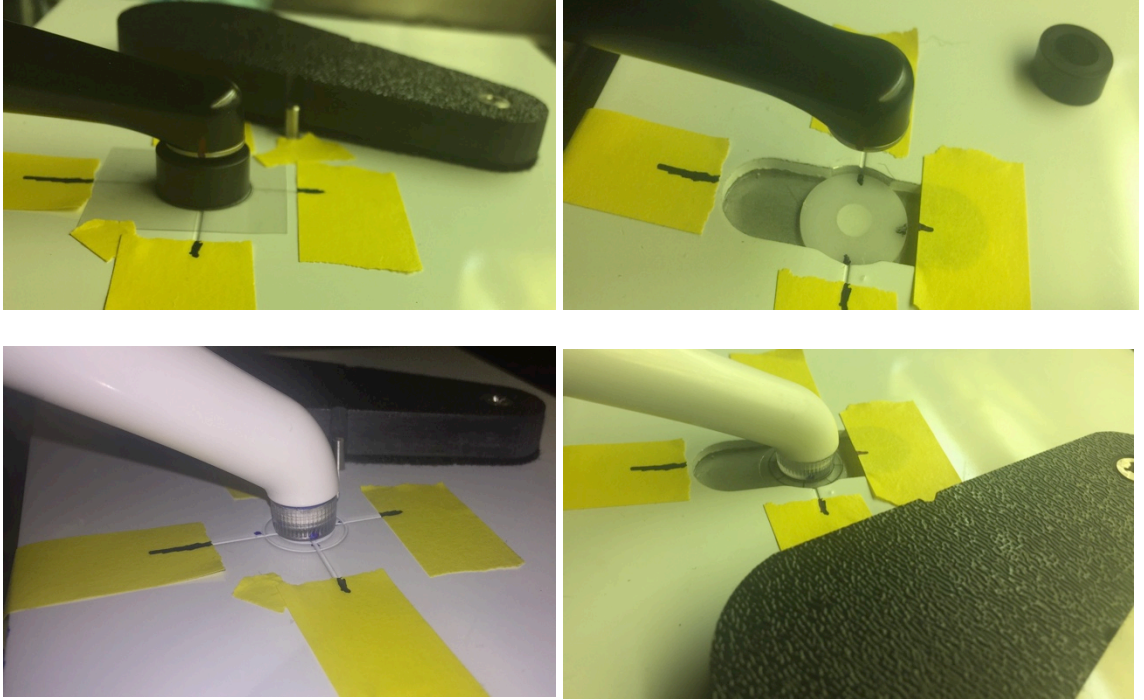


FIGURE-3. Reference points on the MARC-RC system, the rims of LCUs and transparent guide template were used to standardize the positions of LCUs throughout the measuring process.

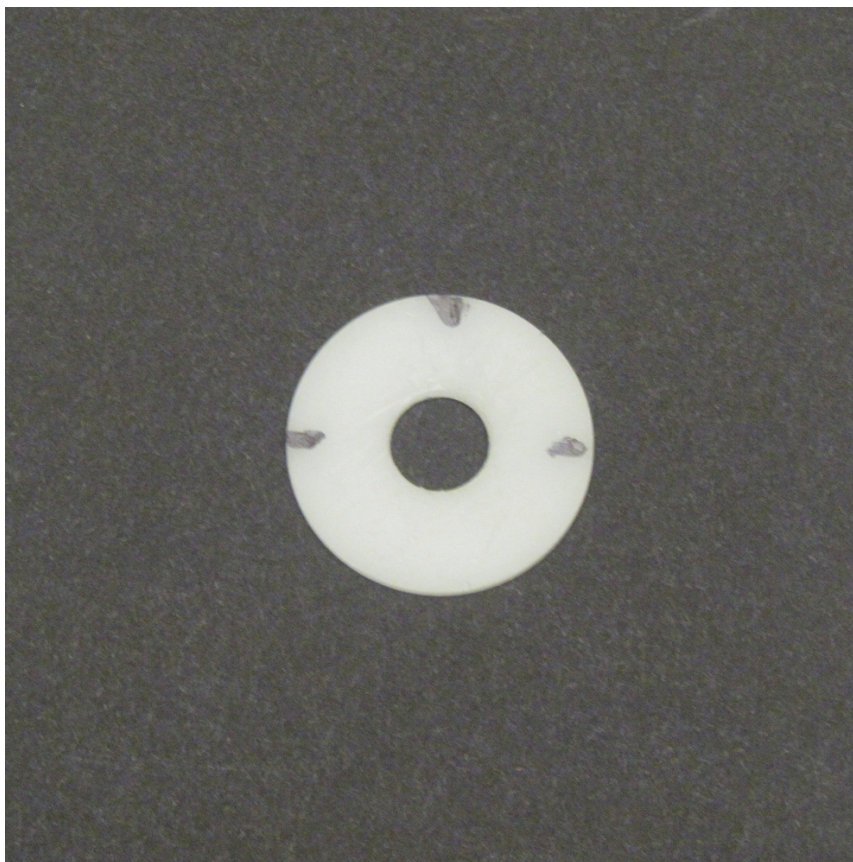


FIGURE-4. Marks on the mold were placed to standardize the location of the sample when performing the DC and microhardness experiments.

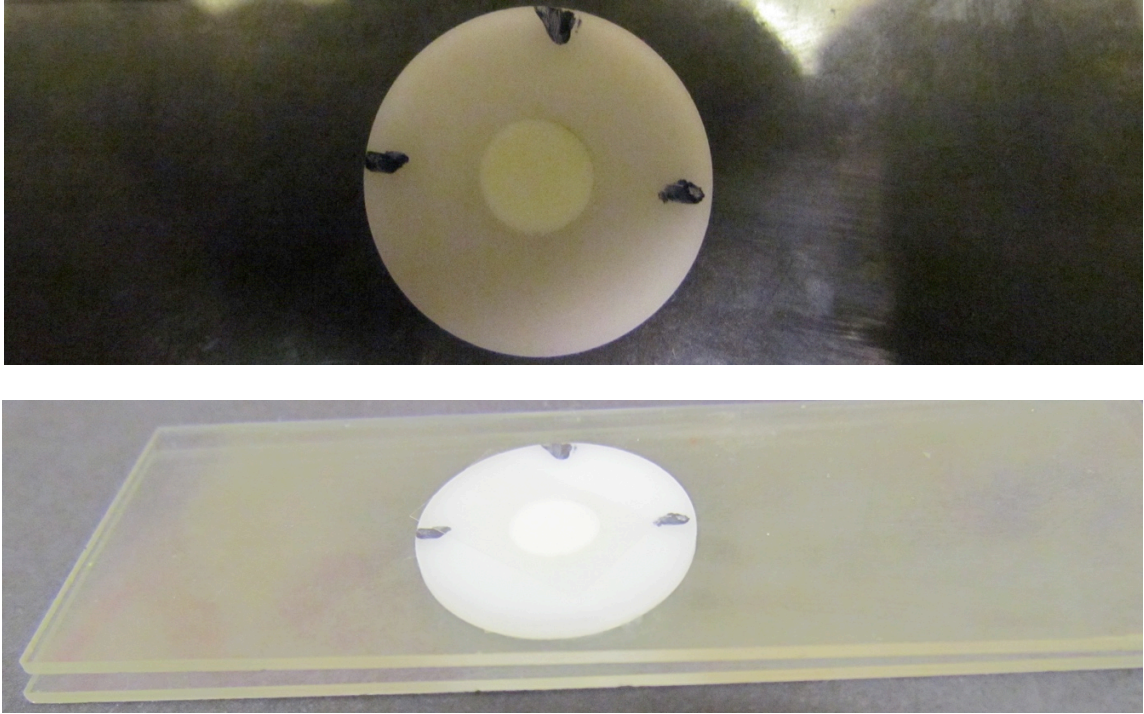


FIGURE-5. Sample mold filled with resin-based pit and fissure sealant after that was placed between two glass slabs to avoid air entrapment.

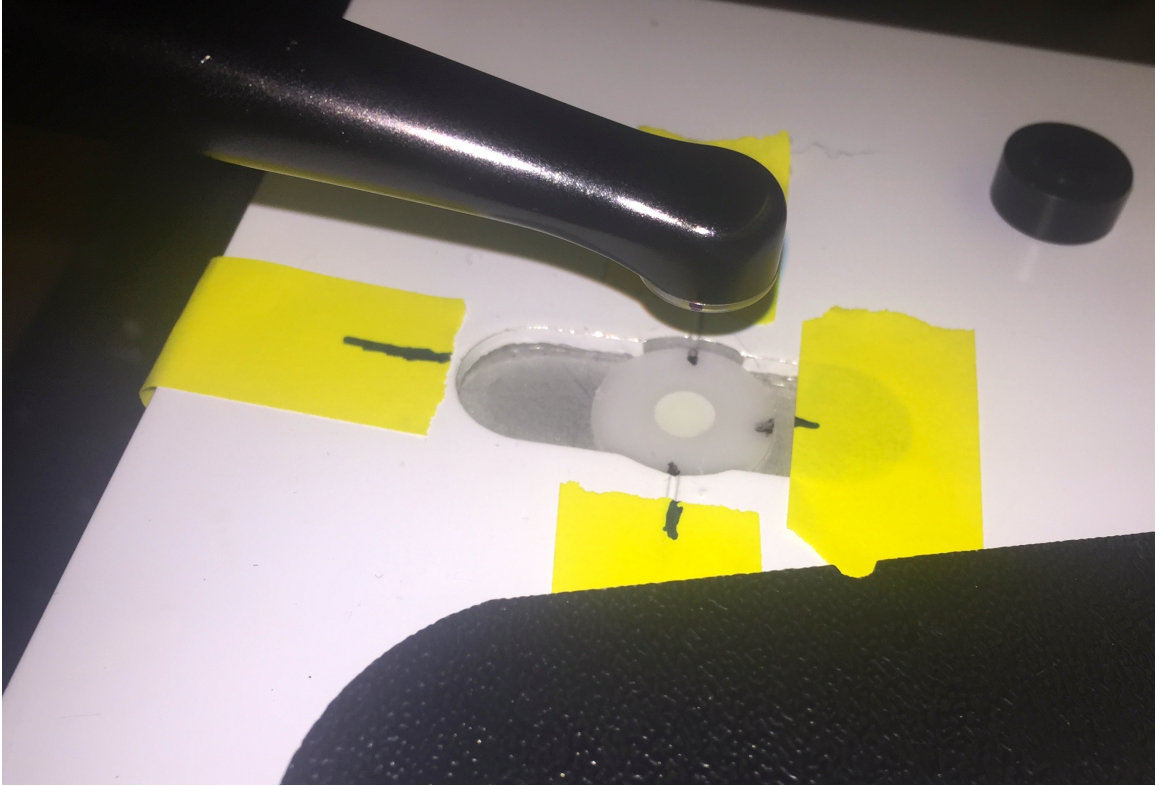


FIGURE-6. The LCU guide tip on the MARC-RC bottom sensor.





FIGURE-7. Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) spectroscopy device.

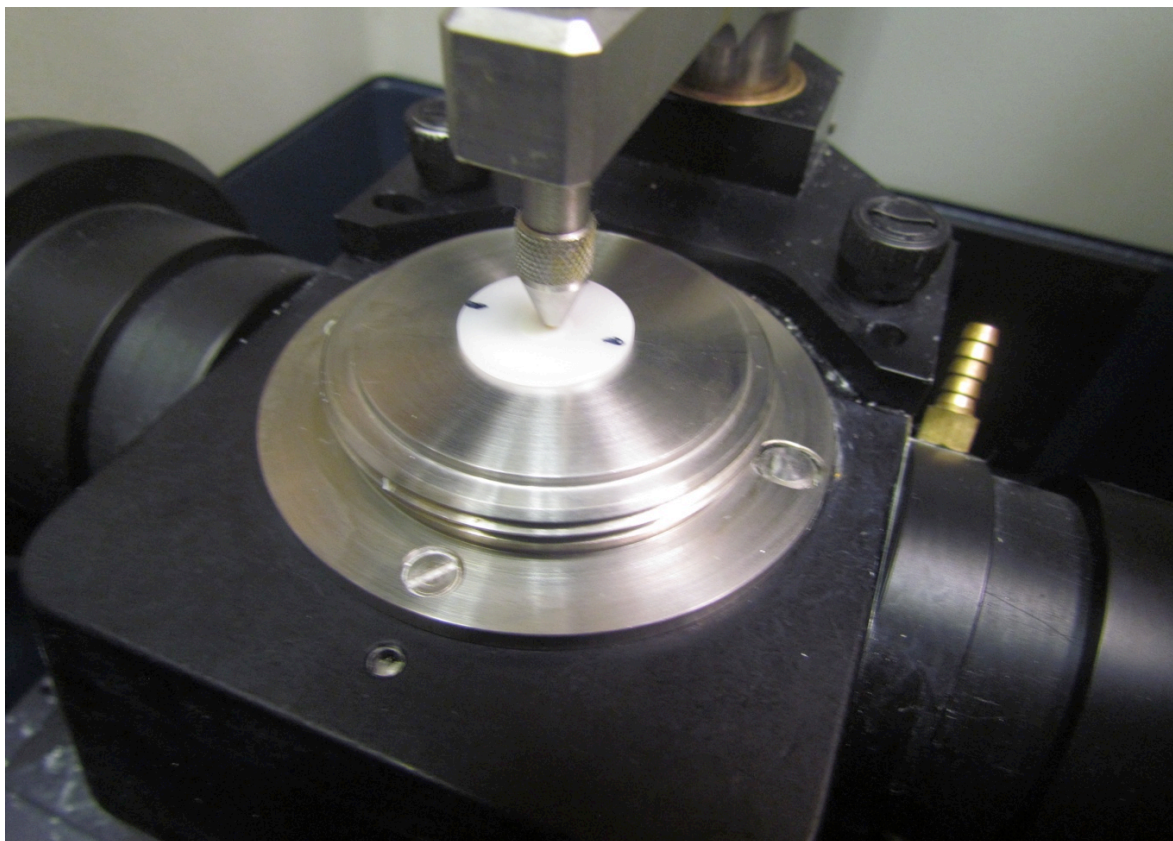


FIGURE-8. The crystal plate and samples secured using a swivel pressure clamp on the FTIR-ATR to stabilize the sample on the crystal.

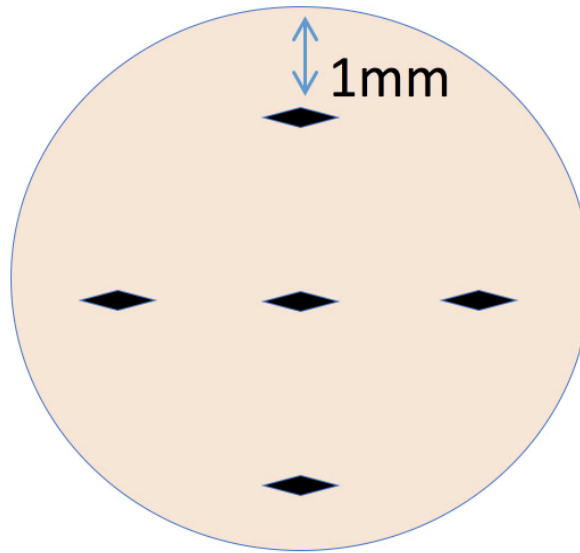


FIGURE-9. Five indentations were located in the upper, lower, left, right, and center of each test surface with the indentations 1mm from the periphery and 2mm between indentations.

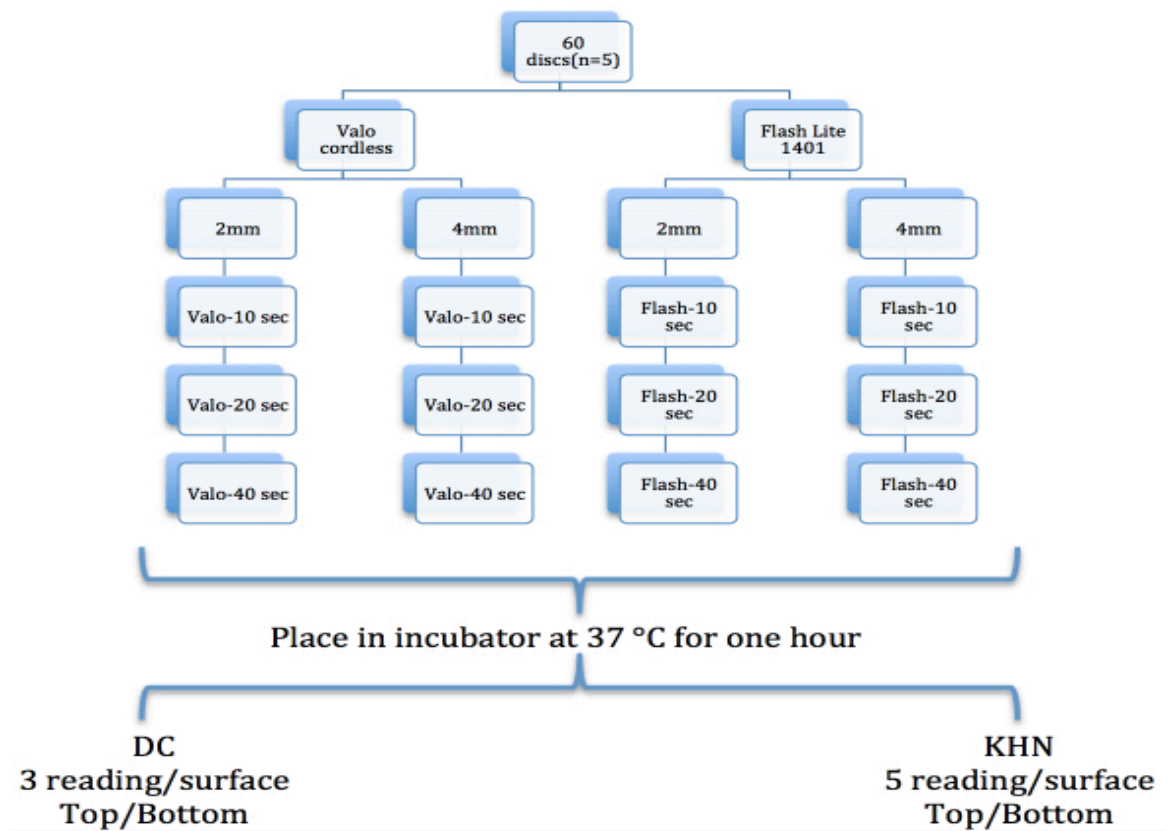


CHART – I. Experiment flow chart.

## DISCUSSION



This study assessed the performance of a multiple emission peak light emitting diode light curing unit (LED LCU) and a single emission peak LED LCU at various light curing times and distances. Polymerization efficiency of a resin-based pit and fissure sealant was evaluated by means of measuring degree of conversion (DC) and microhardness. The three null hypotheses were rejected in the present study. The results showed significant differences in the irradiance, DC and KHN values at three curing times and two curing distances. The multiple emission peak LED LCU showed significantly higher irradiance, microhardness and DC values compared to the single emission peak LED LCU regardless of the curing time or curing distance. This may be explained by the differences in the emitted wavelengths for each LCU as the multiple emission peak LED LCU had two narrow peaks in the range between 395–480 nm while the single emission peak LED LCU was limited to 460–490 nm. The results of this study were consistent with other studies that reported an increased KHN and DC values when comparing the curing performance of single and multiple emission peak LED LCU.<sup>17,116,117</sup>

Two regulating bodies specifying requirements for many dental products are the American National Standards Institute/American Dental Association (ANSI/ADA) and the International Organization for Standardization (ISO). ANSI/ADA specification 39 for pit and fissure sealants requires a 0.75-mm depth of cure<sup>111</sup> while ISO specification 6874 requires a cure twice as deep, 1.5 mm.<sup>112</sup> In a clinical situation, pit and fissure sealants usually have a thickness of 1-mm or less. The light tip of the curing unit may be placed at different distances from the sealant surface.<sup>114</sup> This is mostly dictated by the cusp size and the morphology of pits and fissures, which may lead to increase in the light dispersion and decrease in the irradiance of the light that reaches the material. Therefore, both distance

and sample thickness had to be considered in the present study design to simulate clinical conditions.<sup>102,114</sup> In the present experiment, samples were kept in the dark environment at 37°C under 100% relative humidity for one hour to approximate clinically relevant conditions as heat energy may induce the decomposition of initiators into free radicals or direct excitation of monomer molecules.<sup>115</sup>

The position of the two LCUs was important during the comparison process in the present study. Therefore, an adjustable mechanical arm accessory was used to allow the LCU to be clamped into position, enabling the LCU to be positioned over the sensors and maintain that orientation. That was to stabilize the LCU guide tip in position to make sure the samples received the same amount of energy. Reference points on the MARC-RC system, the rims of the LCUs and a transparent guide template were used to standardize the positions of LCUs throughout the measuring process. The LCU light guide tip was placed perpendicular to and centered on the MARC-RC sensors and the top surface of the specimens. A custom metal ring was used with the multiple emission peak LCU to ensure the guide tip was perpendicular to the sensors because the end of the light guide tip is not flat. The single emission peak LCU guide tip was flat so it was adjusted on the sensors directly within the template marks.

Using short curing times for the resin-based fissure sealant can be advantageous when placing fissure sealants on pediatric patients. In the present study, the resin-based sealant was tested at a 10 second curing time for both LCUs to confirm that this short curing time would not affect the physical and mechanical properties of the resin based fissure sealant.

The radiant exposure values were higher at the 40-second exposure time than at 20 and 10 seconds. (Tables-11, 12) Thus, the increase in exposure time caused an increase in radiant exposure, resulting in higher DC and KHN values at the 40- second exposure time compared to 10 and 20 second on both the top and bottom surfaces. A previous 2005 study by A. Peutzfeldt et al discussed that when the radiant exposure increases, the mechanical properties will be higher. Also, A. Catelan et al stated in 2014 that even without changes in the irradiance the radiant exposure will be higher if exposure time is longer.<sup>128,129</sup>

The test of irradiance, DC and KHN measurements in the present study showed that sealant curing times and light curing distances had a direct influence on the conversion of monomer units into a polymer matrix.<sup>114,134</sup> Moreover, previous studies concluded that inhomogeneous irradiance output from the LCU could result in inhomogeneous polymerization in some areas of the target restoration.<sup>122,98</sup> Increasing the curing distance and/or reducing curing time will lead to a decrease in DC and KHN.<sup>123, 124</sup> Those observations were confirmed in the present experiment. The difference in irradiance between the 2 and 4 mm positions is approximately 400 mW/cm<sup>2</sup> less at the higher position in both LCU. The irradiance, DC and KHN decreased as the distance from the tip of the LCU to the resin-based materials' surface increased because the light intensity was reduced.<sup>104-107</sup> The 2 and 4 mm curing distances were selected due to variations in accessibility, cusp size and shape of posterior teeth, as it may be difficult clinically to place the light tip at 0 mm distance over the resin-based materials surface. Therefore, it is recommended that the distance should not exceed 3 mm to sufficiently cure a 2 mm layer of the composite material.<sup>105</sup> Previous studies found a difference when the distance was less than 4 mm, Rueggeberg et al 1993,<sup>124</sup> or 6 mm, Lindberg A et al. 2004 .<sup>125</sup> The distance

of the light guide of LCU and the thickness of the resin was reported by Price et al 2000 as a factor influencing the energy output.<sup>126</sup> Moreover, from the result of the present study a simple comparison of the difference in the values of DC and KHN between the 10 second and 20 second exposure time showed higher differences than 20 second and 40 second exposure time values. This observation might be explained that the efficiency of the polymerization reaction is limited or reaches a saturated maximum state above which an increase in the irradiance or exposure time no longer leads to a significant increase in DC.<sup>127,118</sup>

Curing efficacy of resin-based pit and fissure sealant can be measured by direct or indirect methods. The direct methods assess the degree of conversion, such as by ATR-FTIR.<sup>118</sup> The indirect method using hardness testing as a parameter for indicating the degree of conversion is widely accepted. A KHN B/T ratio is suggested to verify the efficiency of the cure in deep surfaces when compared to surfaces located closer to the light source.<sup>119</sup> If polymerization is effective, the hardness ratio should be 1, as the hardness of the bottom surface should be the same as the top surface. The difference in the B/T hardness ratio results should not exceed 10% to 20% (KHN B/T ratio  $\geq 0.80$ ) for light activated composites to be adequately polymerized.<sup>120</sup> Therefore, the hardness ratios obtained in this study were less than 0.80 for all irradiation protocols with the Delton Opaque except the 40 second at 2mm curing distance group for both curing units. (Table-10) In the case of a 10 second at 4mm curing time, no microhardness value was obtained for the bottom surfaces due to inadequate polymerization. Those findings support a 2011 study by Duangthip et al.<sup>132</sup> Contradictory findings also have been reported.<sup>121</sup> According to Warnock and Rueggeberg, the second generation LEDs reached a conversion similar to

the control in only 10 seconds.<sup>121</sup> It should be noted, however, that the sealants were tested at only a 0.5-mm-thick layer. This higher conversion could have resulted from less light attenuation of the thinner sealant. In the present study, the 1-mm thickness of sealants may have compromised the hardness ratio, especially because opaque dental sealants were used.

The opacity of the opaque white dental sealant that was used in this study is related to the opacifying agents present in its composition. This probably causes substantial reflection, scattering, and absorption of the light energy, which may prevent a more thorough cure through the sealant. A previous study by Yue et al reported greater depth of cure for Delton Clear than Delton Opaque irrespective of the curing time or distance.<sup>130, 104</sup> As a result, the polymerization reaction is attenuated in an opaque sealant and the DC, KHN of this material is decreased. This may be associated with the presence of titanium oxide fillers in the opaque version of this sealant which may have interfered with light penetration through the material. Moreover, Shortall et al 1995 have attributed this type of effect in composite materials to changes in refractive index mismatch between filler material and resin during the curing time course.<sup>131-134</sup>

The present study had some limitations. The type and amount of photoinitiators included in resin-based pit and fissure sealant that was used is not clearly mentioned. The manufacturers' information didn't not mention the type of photoinitiator that was used in the material. Therefore, further studies will be needed to determine accurately the performance of single and multiple emission peak LED LCUs on resin-based pit and fissure sealant formulated with different concentrations and ratios of CQ and alternative photoinitiators.

## CONCLUSION

Within the bounds of the present study the multiple emission peak LED LCU demonstrated significantly higher irradiance, the DC and the KHN than the single emission peak LED LCU at specific curing distances and curing times. The differences in the values were obvious in the bottom side of the samples. The irradiance, DC and KHN were shown to be significantly influenced by the exposure time, exposure distance and type of curing unit. Based on these findings an exposure time should be encouraged to be at 40 s and an exposure distance should be less than 4 mm, since it can reach the polymerization that would lead to higher mechanical and physical properties at least within the same material and light curing units that were used in the present study. Furthermore, clinicians should be more knowledgeable when they choose these devices and sealant materials that will be matched to achieve optimal polymerization and mechanical properties. In addition, it should be stressed that the findings of this study are valid only for the specific sealant material and LCUs studied; these results cannot be generalized to all sealants and curing protocols. Thus, more studies are needed to clarify the relationship between newer light curing technology and sealant polymerization.

## REFERENCES



1. Wright JT, Tampi MP, Graham L, et al. Sealants for preventing and arresting pit-and-fissure occlusal caries in primary and permanent molars. *Pediatr Dent* 2016; 38(4): 282-308.
2. Beauchamp J, Caufield PW, Crall JJ, et al. Evidence-based clinical recommendations for the use of pit-and-fissure sealants: a report of the American Dental Association Council on Scientific Affairs. *Dent Clin North Am* 2009 Jan; 53(1): 131-47.
3. Ahovuo-Saloranta A, Forss H, Hiiri A, et al. Pit and fissure sealants versus fluoride varnishes for preventing dental decay in the permanent teeth of children and adolescents. *Cochrane Database of Systematic Reviews* 2016 Jan; 18;(1).
4. RJ S. *Clinical Applications of the Acid Etch Technique*. 1st ed. Quintessence Publishing Co, Inc. 1978: 19-42.
5. Cueto EI, Buonocore MG. Adhesive sealing of pits and fissures for caries prevention. *J Dent Res* 1965; 44(137).
6. Soto-Rojas AE, Krushinski C, Eberhardt C, Maupome G. Comparison of Isolation Methods for Sealing Teeth in a Mobile Program. *Caries Res* 2010; 121.
7. Simonsen RJ, Neal RC. A review of the clinical application and performance of pit and fissure sealants. *Aust Dent J* 2011 Jun; 56 Suppl 1:45-58.
8. San-Martin L, Ogunbodede EO, Kalenderian E. A 50-year audit of published peer-reviewed literature on pit and fissure sealants, 1962–2011. *Acta Odontologica Scandinavica* 2013 Nov; 71(6): 1356-61.
9. de Oliveira DC, Rocha MG, Gatti A, Correr AB, Ferracane JL, Sinhoret MA. Effect of different photoinitiators and reducing agents on cure efficiency and color stability of resin-based composites using different LED wavelengths. *Journal of Dentistry* 2015; 43: 2015 Dec;43(12):1565-72.
10. Santini A, Miletic V, Swift MD, Bradley M. Degree of conversion and microhardness of TPO-containing resin-based composites cured by polywave and monowaveLED units. *Journal of Dentistry* 2012 Jul; 40(7):577-84.
11. Aldossary Mohammed, Santini Ario. Resin-Based Composite and LCU-related Factors Affecting the Degree of Cure. A Literature Review:Part 1,2. Resin-Based Composites. *Acta Medica Marisiensis* 2015 sep; 61(3):153-7.
12. Platt JA, Clark H, Moore BK. Curing of Pit & Fissure Sealants Using Light Emitting Diode Curing Units. *Operative Dentistry* 2005 Nov-Dec; 30(6):764-71.
13. Platt JA. Light Curing Explored in Halifax. *Operative Dentistry* 2014 Nov-Dec; 39(6):561-3.

14. Turssi CP, Ferracane JL, Vogel K. Filler features and their effects on wear and degree of conversion of particulate dental resin composites. *Biomaterials J* 2005 Aug; 26(24):4932-7.
15. Manojlovic D, Radisic M, Vasiljevic T, Zivkovic S, Lausevic M, Miletic V. Monomer elution from nano- hybrid and ormocer-based composites cured with different light sources. *Dent Mater* 2011 Apr; 27(4):371-8.
16. Guiraldo RD, Consani S, Sinhoreti MA, Correr-Sobrinho L, Schneider LF. Thermal variations in the pulp chamber associated with composite insertion techniques and light-curing methods. *Journal of Contemporary Dental Practice* 2009 Jan 1; 10(1):17-24.
17. Leprince J, Devaux J, Mullier T, Vreven J, Leloup G. Pulpal-temperature rise and polymerization efficiency of LED curing lights. *Operative Dentistry* 2010 Mar-Apr; 35(2):220-30.
18. Cassoni A, Ferla Jde O, Shibli JA, Kawano Y. Knoop microhardness and FT-Raman spectroscopic evaluation of a resin-based dental material light-cured by an argon ion laser and halogen lamp. *Photomed Laser Surg* 2008 Dec; 26(6): 531-9.
19. Kühnisch J1, Mansmann U, Heinrich-Weltzien R, Hickel R. Longevity of materials for pit and fissure sealing--results from a meta-analysis. *Dent Mater.* 2012 Mar; 28(3):298-303.
20. Stansbury JW, Dickens SH. Determination of double bond conversion in dental resins by near infrared spectroscopy. *Dental Materials J* 2001 Jan;17(1):71-9.
21. Ferracane JL. Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. *Dental Materials J* 1985 Feb; 1(1):11-4.
22. Castillo Dutra Borges B, Roger Pinho de Silva P, Catelan A, Henrique Baggio Aguiar F. Influence of the Light Curing Tip Distance and Material Opacity on Selected Physical Properties of a Pit and Fissure Sealant. *Pediatric Dentistry J* 2011 Nov-Dec; 33(7):505-9.
23. Yan YL, Kim YK, Kim KH, Kwon TY. Changes in degree of conversion and microhardness of dental resin cements. *Operative Dentistry* 2010 Mar-Apr; 35(2): 203-10. doi: 10.2341/09-174-L.
24. Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. *Clinical Oral Investigations* 2013 Jan; 17(1): 227-35. doi: 10.1007/s00784-012-0702-8. Epub 2012 Mar 14.
25. Smallridge J; Faculty of Dental Surgery, Royal College of Surgeons. UK National Clinical Guidelines in Paediatric Dentistry. Management of the stained fissure in the first permanent molar. *Int J Paediatr Dent* 2000 Mar; 10(1):79-83.

26. Kenneth J. Anusavice. Phillips' Science of Dental Materials. St Louis, MO: Elsevier/Saunders 2013.
27. C. Splieth, M. Förster, G. Meyer. Additional caries protection by sealing permanent first molars compared to fluoride varnish applications in children with low caries prevalence: a 2-year results. *Eur J Paediatr Dent* 2001 sep; 2(3):133-7.
28. JL F. Current trends in dental composites. *Crit Rev Oral Biol Med* 1995; 6:302-18.
29. Glance CLaa. LED Technology Here to stay 2002. 3m ESPE. 2002:1-6.
30. Jandt KD, Mills RW. A brief history of LED photopolymerization. *Dental Materials Journal* 2013 Jun; 29(6):605-17.
31. Leonard DL, Charlton DG, Roberts HW, Cohen ME. Polymerization efficiency of LED curing lights. *J Esthet Restor Dent* 2002; 14(5):286-95.
32. Christensen GJ. The curing light dilemma. *J Am Dent Assoc* 2002; 133:761-3.
33. Hofmann N, Hugo B, Klaiber B. Effect of irradiation type (LED or QTH) on photo-activated composite shrinkage strain kinetics, temperature rise, and hardness. *Eur J Oral Sci.* 2002 Dec; 110(6):471-9.
34. Jandt KD, Mills RW, Blackwell GB, Ashworth SH. Depth of cure and compressive strength of dental composites cured with blue light emitting diodes (LEDs). *Dent Mater* 2000 Jan; 16(1): 41-7.
35. Kitchens B, Wells M, Tantbirojn D, Versluis A. Depth of cure of sealants polymerized with high-power light emitting diode curing lights. *International Journal of Paediatric Dentistry* 2015 Mar; 25(2):79-86.
36. Asmussen E, Peutzfeldt A. Influence of specimen diameter on the relationship between subsurface depth and hardness of a light-cured resin composite. *Eur J Oral Sci* 2003 Dec; 111(6): 543-6.
37. Obici AC, Sinhoreti MA, Correr Sobrinho L, de Goes MF, Consani S. Evaluation of depth of cure and knoop hardness in a dental composite photoactivated using different methods. *Braz Dent J* 2004; 15(3): 199-203. Epub 2005 Mar 18.
38. Leprince JG, Palin WM, Hadis MA, Devaux J, Leloup G. Progress in dimethacrylate-based dental composite technology and curing efficiency. *Dental Materials* 2013 Feb; 29(2): 139-56.
39. Schmalz, Gottfried, Arenholt Bindslev, Dörthe. Resin-based composites. Biocompatibility of Dental Materials. Berlin/Heidelberg/Germany: Springer-Verlag 2009:99–137.

40. Cramer NB, Stansbury JW, Bowman CN. Recent advances and developments in composite dental restorative materials. *Journal of Dental Research* 2011 Apr; 90(4): 402-16.
41. K. S. Anseth, S. M. Newman, C. N. Bowman. Polymeric dental composites: properties and reaction behavior of multimethacrylate dental restorations. *Advances in Polymer Science* 1995; 122:177–217.
42. Lovell LG, Newman SM, Bowman CN. Effects of composition and reactivity on the reaction kinetics of dimethacrylate/dimethacrylate copolymerizations. *J Dent Res* 1999 Aug; 78(8): 1469-76.
43. Truffier-Boutry D, Demoustier-Champagne S, Devaux J, Biebuyck JJ, Mestdagh M, Larbanois P, Leloup G. A physico-chemical explanation of the post-polymerization shrinkage in dental resins. *Dental Materials* 2006 May; 22(5): 405-12.
44. Ferracane JL, Greener EH. The effect of resin formulation on the degree of conversion and mechanical properties of dental restorative resins. *Journal of Biomedical Materials Research* 1986 Jan; 20(1): 121-31.
45. Li J, Li H, Fok AS, Watts DC. Multiple correlations of material parameters of light-cured dental composites. *Dental Materials* 2009 Jul; 25(7): 829-36.
46. Dewaele M, Truffier-Boutry D, Devaux J, Leloup G. Volume contraction in photocured dental resins: the shrinkage–conversion relationship revisited. *Dental Materials* 2006 Apr; 22(4): 359-65.
47. Ferracane JL, Mitchem JC, Condon JR, Todd R. Wear and marginal breakdown of composites with various degrees of cure. *Journal of Dental Research* 1997 Aug; 76(8): 1508-16.
48. Ferracane JL. Elution of leachable components from composites. *Journal of Oral Rehabilitation* 1994 Jul; 21(4): 441-52.
49. Ferracane JL, Greener EH. Fourier transform infrared analysis of degree of polymerization in unfilled resins – methods comparison. *Journal of Dental Research* 1984 Aug; 63(8): 1093-5.
50. Pianelli C, Devaux J, Bebelman S, Leloup G. The micro-Raman spectroscopy, a useful tool to determine the degree of conversion of light-activated composite resins. *Journal of Biomedical Materials Research* 1999; 48(5): 675-81.
51. Duangthip D1, Ballungpattama S, Sitthisettapong T. Effect of Light Curing Methods on Microleakage and Microhardness of Different Resin Sealants. *Journal of Dentistry for Children* 2011 Jul; 78(2): 88-95.

52. Ferracane JL. Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. *Dental Materials* 1985 Feb; 1(1): 11-4.
53. Leprince JG, Leveque P, Nysten B, Gallez B, Devaux J, Leloup G. New insight into the “depth of cure” of dimethacrylate-based dental composites. *Dental Materials* 2012 May; 28(5): 512-20.
54. Musanje L, Darvell BW. Curing-light attenuation in filled–resin restorative materials. *Dental Materials* 2006 Sep; 22(9): 804-17.
55. Dewaele M, Asmussen E, Peutzfeldt A, Munksgaard EC, Benetti AR, Finné G, Leloup G, Devaux J. Influence of curing protocol on selected properties of light-curing polymers: Degree of conversion, volume contraction, elastic modulus, and glass transition temperature. *Dental Materials* 2009 Dec; 25(12): 1576-84.
56. Palin WM, Fleming GJ, Marquis PM. The reliability of standardized flexure strength testing procedures for a light-activated resin-based composite. *Dent Materials* 2005 Oct; 21(10): 911-9.
57. Ilie N, Hickel R, Watts DC. Spatial and cure-time distribution of dynamic-mechanical properties of a dimethacrylate nano-composite. *Dental Materials* 2009 Mar; 25(3): 411-8.
58. Flury S, Hayoz S, Peutzfeldt A, Hüsler J, Lussi A. Depth of cure of resin composites. is the ISO 4049 method suitable for bulk fill materials?. *Dental Materials* 2012 May; 28(5): 521-8.
59. Leprince J, Lamblin G, Truffier-Boutry D, Demoustier-Champagne S, Devaux J, Mestdagh M, Leloup G. Kinetic study of free radicals trapped in dental resins stored in different environments. *Acta Biomaterialia* 2009 Sep; 5(7):2518-24.
60. WD C. Photopolymerization kinetics of dimethacrylates using the camphorquinone amine initiator system. *Polymer* 1992; 33: 600–9.
61. Jakubiak J AX, Fouassier JP, Sionkowska A, Andrzejewska E, Linden LA. Camphorquinone-amines photoinitiating systems for the initiation of free radical polymerization. *Polymer* 2003; 44:5219–26.
62. Musanje L, Ferracane JL, Sakaguchi RL. Determination of the optimal photoinitiator concentration in dental composites based on essential material properties. *Dental Materials* 2009 Aug; 25(8): 994-1000.
63. Pfeifer CS, Ferracane JL, Sakaguchi RL, Braga RR. Photoinitiator content in restorative composites: influence on degree of conversion, reaction kinetics, volumetric shrinkage and polymerization stress. *American Journal of Dentistry* 2009 Aug; 22(4): 206-10.

64. Shin DH, Rawls HR. Degree of conversion and color stability of the light curing resin with new photoinitiator systems. *Dental Materials* 2009 Aug; 25(8):1030-8.
65. Park J, Ye Q, Topp EM, Misra A, Kieweg SL, Spencer P. Effect of photoinitiator system and water content on dynamic mechanical properties of a light-cured bisGMA/HEMA dental resin. *Journal of Biomedical Materials Research – Part A* 2010 Jun 15; 93(4): 1245-51.
66. Wayne D. Cook, Fei Chen. Enhanced photopolymerization of dimethacrylates with ketones, amines, and iodonium salts: the CQ system. *Journal of Polymer Science Part A: Polymer Chemistry* 2011 Dec; 49(23): 5030 - 5041.
67. Lovell LG, Newman SM, Bowman CN. The effects of light intensity, temperature, and comonomer composition on the polymerization behavior of dimethacrylate dental resins. *Journal of Dental Research* 1999 Aug; 78(8): 1469-76.
68. Ogunyinka A, Palin WM, Shortall AC, Marquis PM. Photoinitiation chemistry affects light transmission and degree of conversion of curing experimental dental resin composites. *Dental Materials* 2007 Jul; 23(7): 807-13.
69. Shortall AC, Wilson HJ, Harrington E. Depth of cure of radiation-activated composite restoratives – influence of shade and opacity. *Journal of Oral Rehabilitation* 1995 May; 22(5): 337-42.
70. Watts DC, Cash AJ. Analysis of optical transmission by 400–500 nm visible light into aesthetic dental biomaterials. *Journal of Dentistry* 1994 Apr; 22(2): 112-7.
71. Musanje L, Darvell BW. Curing-light attenuation in filled–resin restorative materials. *Dental Materials* 2006 Sep; 22(9): 804-17.
72. Chen YC, Ferracane JL, Prahl SA. Quantum yield of conversion of the photoinitiator camphorquinone. *Dental Materials* 2007 Jun; 23(6): 655-64.
73. E. Lienhard. instrument for transmitting ultra-violet radiation to a limited area. *United States Patent* 1973; 3(712): 984.
74. Craig RG. Chemistry composition, and properties of composite resins. In: Horn H, editor. *Symposium on composite resins in dentistry*. Philadelphia, Saunders: The Dental Clinics of North America 1981 Apr; 25(2): 219-39.
75. Stansbury JW. Curing dental resins and composites by photopolymerization. *Esthet Dent* 2000; 12(6): 300-8.
76. Donald I. Gonser. Xenon light apparatus for supplying ultraviolet and visible spectra. *United States Patent* 1980; 4:229,658.

77. Tirtha R, Fan PL, Dennison JB, Powers JM. In vitro depth of cure of photo-activated composites. *J Dent Res* 1982 Oct; 61(10): 1184-7.
78. Wilson DN. Personal communication. 2008.
79. Rueggeberg FA. State-of-the-art: Dental photocuring—A review. *Dental Materials* 2011; 27: 39–52.
80. Krämer N, Lohbauer U, García-Godoy F, Frankenberger R. Light curing of resin-based composites in the LED era. *American Journal of Dentistry* 2008 Jun; 21(3): 135-42.
81. Price RB, Felix CA. Effect of delivering light in specific narrow bandwidths from 394 to 515 nm on the microhardness of resin composites. *Dental Materials* 2009 Jul; 25(7): 899-908.
82. Price RB, Labrie D, Rueggeberg FA, Felix CM. Irradiance uniformity and distribution from dental light curing units. *Journal of Esthetic and Restorative Dentistry* 2010 Dec; 22(6): 363-77.
83. Arikawa H, Takahashi H, Minesaki Y, Muraguchi K, Matsuyama T, Kanie T, Ban S. A method for improving the light intensity distribution in dental light-curing units. *Dental Materials Journal* 2011; 30(2): 151-7.
84. Nomoto R, McCabe JF, Nitta K, Hirano S. Relative efficiency of radiation sources for photopolymerization. *Odontology* 2009 Jul; 97(2): 109-14.
85. Miyazaki M, Oshida Y, Moore BK, Onose H. Effect of light exposure on fracture toughness and flexural strength. *Dent Mater J* 1996 Nov; 12(6): 328-32.
86. Price RB, Felix CA, Andreou P. Effects of resin composite composition and irradiation distance on the performance of curing lights. *Biomaterials J* 2004 Aug; 25(18): 4465-77.
87. Price RB, Fahey J, Felix CM. Knoop hardness of five composites cured with single-peak and polywave LED curing lights. *Quintessence International* 2010 Nov-Dec; 41(10): e181-91.
88. Arikawa H, Kanie T, Fujii K, Takahashi H, Ban S. Effect of inhomogeneity of light from light curing units on the surface hardness of composite resin. *Dental Materials Journal* 2008 Jan; 27(1): 21-8.
89. Vandewalle KS, Roberts HW, Rueggeberg FA. Power distribution across the face of different light guides and its effect on composite surface microhardness. *Journal of Esthetic and Restorative Dentistry* 2008; 20(2): 108-17; discussion 118.

90. Vandewalle KS. Irradiance differences in the violet (405 nm) and blue (460 nm) spectral ranges among dental light-curing units. *Journal of Esthetic and Restorative Dentistry* 2010 Dec; 22(6): 378.
91. Vandewalle KS, Roberts HW, Andrus JL, Dunn WJ. Effect of light dispersion of LED curing lights on resin composite polymerization. *Journal of Esthetic and Restorative Dentistry* 2005; 17(4): 244-54; discussion 254-5.
92. Ilie N, Jelen E, Hickel R. Is the soft-start polymerisation concept still relevant for modern curing units?. *Clinical Oral Investigations* 2011 Feb; 15(1): 21-9.
93. Kanca J 3rd, Suh BI. Pulse activation: reducing resin-based composite contraction stresses at the enamel cavosurface margins. *American Journal of Dentistry* 1999 Jun; 12(3): 107-12.
94. Cunha LG, Alonso RC, Pfeifer CS, Correr-Sobrinho L, Ferracane JL, Sinhoreti MA. Contraction stress and physical properties development of a resin-based composite irradiated using modulated curing methods at two C-factor levels. *Dental Materials* 2008 Mar; 24(3): 392-8.
95. Cunha LG, Alonso RC, Pfeifer CS, Correr-Sobrinho L, Ferracane JL, Sinhoreti MA. Modulated photoactivation methods: Influence on contraction stress, degree of conversion and push-out bond strength of composite restoratives. *Journal of Dentistry* 2007 Apr; 35(4): 318-24.
96. Price RB, Whalen JM, Price TB, Felix CM, Fahey J. The effect of specimen temperature on the polymerization of a resin-composite. *Dental Materials* 2011 Oct; 27(10): 983-9.
97. Michaud PL, Price RB, Labrie D, Rueggeberg FA, Sullivan B. Localised irradiance distribution found in dental light curing units. *Journal of dentistry* 2014 Feb; 42(2): 129-39.
98. Daronch M, Rueggeberg FA, De Goes MF, Giudici R. Polymerization kinetics of pre-heated composite. *Journal of Dental Research* 2006 Jan; 85(1): 38-43.
99. Price RB, Labrie D, Whalen JM, Felix CM. Effect of distance on irradiance and beam homogeneity from 4 light-emitting diode curing units. *Journal of Canadian Dental Association* 2011; 77:b9.
100. Fróes-Salgado NR, Pfeifer CS, Francci CE, Kawano Y. Influence of photoactivation protocol and light guide distance on conversion and microleakage of composite restorations. *Operative Dentistry* 2009 Jul-Aug; 34(4): 408-14.
101. Price RB, Felix CM, Whalen JM. Factors affecting the energy delivered to simulated class I and class V preparations. *Journal of Canadian Dental Association* 2010; 76:a94.



102. Mills RW, Uhl A, Jandt KD. Optical power outputs, spectra and dental composite depths of cure, obtained with blue light emitting diode (LED) and halogen light curing units (LCUs). *Br Dent J* 2002 Oct 26; 193(8): 459-63; discussion 455.
103. Ikemura K, Endo T. A review of the development of radical photopolymerization initiators used for designing light-curing dental adhesives and resin composites. *Dental Materials Journal* 2010 Oct; 29(5): 481-501.
104. Zhu S, Platt J. Curing efficiency of three different curing modes at different distances for four composites. *Operative Dentistry Journal* 2011 Jul-Aug; 36(4): 362-71.
105. Rode KM, Kawano Y, Turbino ML. Evaluation of curing light distance on resin composite microhardness and polymerisation. *Operative Dentistry Journal* 2007 Nov-Dec; 32(6): 571-8.
106. Pires JA, Cvitko E, Denehy GE, Swift EJ Jr. Effects of curing tip distance on light intensity and composite resin microhardness. *Quintessence International* 1993 Jul; 24(7): 517-21.
107. da Silva EM, Poskus LT, Guimarães JG, de Araújo Lima Barcellos A, Fellows CE. Influence of light polymerisation modes on degree of conversion and crosslink density of dental composites. *J Mater Sci Mater Med* 2008 Mar; 19(3): 1027-32.
108. Flury S, Lussi A, Hickel R, Ilie N. Light curing through glass ceramics with a second- and a third-generation LED curing unit: effect of curing mode on the degree of conversion of dual-curing resin cements. *Clin Oral Investig* 2013 Dec; 17(9): 2127-37.
109. Price RB, Felix CA, Andreou P. Third-generation vs a second-generation LED curing light: effect on Knoop microhardness. *Compend Contin Educ Dent* 2006 Sep; 27(9): 490-6; quiz 497, 518.
110. Price RB, Felix CA, Andreou P. Evaluation of a dual peak third generation LED curing light. *Compend Contin Educ Dent* 2005 May; 26(5): 331-2, 334, 336-8 passim; quiz 348.
111. Association AD. ANSI/ADA specification no. 39-1992. Pit and Fissure Sealants. Chicago, Ill: ADA. 1992.
112. Standardization Of. ISO 6874. Dental Resin-based Pit and Fissure Sealants. Geneva, Switzerland: ISO. 1988.
113. Borges BC, Souza-Júnior EJ, Catelan A, Lovadino JR, Dos Santos PH, Paulillo LA, Aguiar FH. Influence of the extended light exposure time on the degree of conversion and plasticization of materials used as pit and fissure sealants. *J Investig Clin Dent* 2010 Nov; 1(2): 151-5.

114. Borges BC, Bezerra GV, Mesquita Jde A, Pereira MR, Aguiar FH, Santos AJ, Pinheiro IV. Effect of irradiation times on the polymerization depth of contemporary fissure sealants with different opacities. *Braz Oral Res* 2011 Mar-Apr; 25(2): 135-42.
115. Gowariker VR VN, Sreedhar J. *Polymer Science*. New Delhi: New Age International. 2005.
116. Miletic V, Santini A. Micro-Raman spectroscopic analysis of the degree of conversion of composite resins containing different initiators cured by polywave or monowave LED units. *J Dent Res* 2012 Feb; 40(2): 106-13.
117. Park H SS, Hur B, Kim H, Kwon Y, Park J. Effect of the difference in spectral outputs of the single and dual-peak LEDs on the microhardness and the color stability of resin composites. *J Korean Acad Conserv Dent* 2011; 36:108.
118. Rueggeberg FA, Craig RG. Correlation of parameters used to estimate monomer conversion in a light-cured composite. *J Dent Res* 1988 Jun; 67(6):932-7.
119. Yap AU, Soh MS, Siow KS. Post-gel shrinkage with pulse activation and soft-start polymerization. *Operative Dentistry Journal* 2002 Jan-Feb; 27(1): 81-7.
120. Pilo R, Cardash HS. Post-irradiation polymerization of different anterior and posterior visible lightactivated resin composites. *Dental Materials* 1992; 8:299-304.
121. Warnock RD, Rueggeberg FA. Curing kinetics of a photo-polymerized dental sealant. *AM J Dent* 2004 Dec; 17(6): 457-61.
122. Rueggeberg FA, Caughman WF, Curtis JW Jr. Effect of light intensity and exposure duration on cure of resin composite. *Operative Dentistry* 1994 Jan-Feb; 19(1): 26-32.
123. Caughman WF, Rueggeberg FA, Curtis JW Jr. Clinical guidelines for photocuring restorative resins. *Journal of the American Dental Association* 1995 Sep; 126(9): 1280-2, 1284, 1286.
124. Rueggeberg FA, Jordan DM. Effect of light-tip distance on polymerization of resin composite. *International Journal of Prosthodontics* 1993 Jul-Aug; 6(4): 364-70.
125. Lindberg A, Peutzfeldt A, van Dijken JW. Curing depths of a universal hybrid and a flowable resin composite cured with quartz tungsten halogen and light-emitting diode units. *Acta Odontologica Scandinavica* 2004 Apr; 62(2): 97-101.
126. Price RB, Murphy DG, Dérand T. Light energy transmission through cured resin composite and human dentin. *Quintessence International* 2000 Oct; 31(9): 659-67.
127. Musanje L, Darvell BW. Polymerization of resin composite restorative materials: exposure reciprocity. *Dental Materials* 2003 Sep; 19(6): 531-41.

128. Peutzfeldt A, Asmussen E. Resin Composite Properties and Energy Density of Light Cure. *J Dent Res* 2005 Jul; 84(7): 659-62.
129. Catelan A, Mainardi Mdo C, Soares GP, de Lima AF, Ambrosano GM, Lima DA, Marchi GM, Aguiar FH. Effect of light curing protocol on degree of conversion of composites. *Acta Odontologica Scandinavica* 2014 Nov; 72(8): 898-902.
130. Yue C, Tantbirojn D, Grothe RL, Versluis A, Hodges JS, Feigal RJ. The depth of cure of clear versus opaque sealants as influenced by curing regimens. *J Am Dent Assoc* 2009; 140:331-8.
131. Shortall AC, Palin WM, Burtscher P. Refractive index mismatch and monomer reactivity influence composite curing depth. *Journal of Dental Research* 2008 Jan; 87(1): 84-8.
132. Duangthip D, Ballungpattama S, Sitthisettapong T. Curing methods on microhardness and microleakage of sealants *Journal of Dentistry for Children* 2011 Jul; 78(2): 88-95.
133. Castillo Dutra Borges B, Roger Pinho de Silva P, Catelan A, Henrique Baggio Aguiar F. Influence of the Light Curing Tip Distance and Material Opacity on Selected Physical Properties of a Pit and Fissure Sealant. *Pediatric Dentistry* 2011 Nov-Dec; 33(7): 505-9.
134. Leprince JG, Palin WM, Hadis MA, Devaux J, Leloup G. Progress in dimethacrylate-based dental composite technology and curing efficiency. *Dent Mater.* 2013 Feb; 29 (2) : 139-56.

## ABSTRACT

**Objective:** The objective was to assess a multiple emission peak light-emitting-diode (LED) light-curing unit (LCU) by measuring the polymerization efficiency through the degree of conversion (DC) and Knoop microhardness (KHN) of a resin-based pit and fissure sealant at various light curing times and two distances compared to a single emission peak LED LCU.

**Method:** Sixty disks of resin-based pit and fissure sealant (Delton, DENTSPLY, York, PA) samples (6x1mm) were fabricated (n=5/LCU/group). Prepared samples were polymerized using 10, 20 and 40 second curing time at 2 or 4 mm curing distances. The irradiance and radiant exposure received on the top/bottom surfaces of the samples were measured using the Managing Accurate Resin Curing-Resin Calibrator (MARC-RC) system. The samples were stored at 37°C for one hour. Then, the DC (n=3/surface) and KHN (n=5/surface) measurements were collected on the top and bottom surfaces using Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR) and a microhardness tester (Instron) utilizing 25-gm at 10 seconds dwell time, respectively. Multiple-way ANOVA was performed followed by Tukey test ( $\alpha=0.05$ ).

**Result:** The irradiance from the multiple emission peak LED LCU was significantly higher than the single emission peak LED LCU (1312.6 and 768.3 mW/cm<sup>2</sup>) respectively. Moreover, the multiple emission peak LED LCU displayed significantly higher DC (82.5%) and microhardness (26.2 KHN) compared to the single emission peak LED LCU (75.5% DC and 21.2 KHN) when curing samples at 2 and 4 mm curing distances assessed using 10, 20 and 40-second curing times. The 10 second cure at 4 mm showed significantly lower DC and KHN values compared to the other groups.

**Conclusion:** The multiple emission peak LED LCU demonstrated significantly higher irradiance, DC and KHN compared to the single emission peak LED LCU on a resin-based pit and fissure sealant at 2 and 4 mm curing distances and 10, 20 and 40 second curing times. Therefore, the multiple emission peak LED LCU performed higher than the single emission peak LED LCU.

## CURRICULUM VITAE

Saleh Ali M Alqahtani

February 1975	Born in Abha, Kingdom of Saudi Arabia
2005-2010	Graduated from King Khalid University College of Dentistry Oral and Dental Surgery (BDS)
August 2010-August 2011	Internship, King Khalid University College of Dentistry.
August 2011-September 2012	Teaching Assistant, Faculty of Dentistry, King Khalid University College of Dentistry.
2013-2014	Graduated from International Comprehensive Dentistry Program One-year program – New York University.
July 2014-July 2017	Certificate in Operative Dentistry Indiana University School of Dentistry Indianapolis, IN.
Professional Organization	Academy of Operative Dentistry Member /International Association of Dental Research (IADR)